

Note: This method is currently a draft. Comments are welcome but must be in writing. Please send comments to M. Papp, US EPA, MD-14, RTP, NC, 27711 (e-mail: papp.michael@epamail.epa.gov). Please identify each comment by section, subsection, and page number

1.0 Scope and Applicability

1.1 Introduction and Purpose

This document is intended to assist personnel of air monitoring agencies that use reference methods or Class I equivalent methods to monitor ambient air for particles with an aerodynamic diameter equal to or less than 2.5 μm , known as $\text{PM}_{2.5}$. The formal specifications for $\text{PM}_{2.5}$ reference method samplers and for obtaining valid reference method measurements are set forth in the Environmental Protection Agency's (EPA's) monitoring regulations at 40 CFR Part 50, Appendix L (EPA 1997a).^{*} This document reviews those formal requirements and provides clarifications and supplemental information in greater detail than can be provided in the formal regulatory requirements. Because this supplemental information is intended for method users, the emphasis is on the operational aspects of the method, rather than on the design and performance specifications for the sampler, which are of more concern to the sampler manufacturer. This document also provides recommended quality assurance (QA) procedures and guidance to help monitoring agencies reliably achieve the data quality objectives (DQOs) established for $\text{PM}_{2.5}$ monitoring. The information provided here, together with the more sampler-specific information and instructions provided by the manufacturer of the selected $\text{PM}_{2.5}$ sampler and contained in the sampler's operation or instruction manual, should be used by each monitoring agency to develop its own agency-specific standard operating procedure (SOP) to govern its individual $\text{PM}_{2.5}$ monitoring activity.

1.2 Applicability

This document is primarily applicable to $\text{PM}_{2.5}$ ambient air monitoring with reference methods carried out by State and local air monitoring agencies in their State and local air monitoring stations (SLAMS) air surveillance networks under the air monitoring requirements of 40 CFR Part 58 (EPA 1997b). The document is also applicable to other organizations required to carry out SLAMS or SLAMS-related $\text{PM}_{2.5}$ monitoring and to any $\text{PM}_{2.5}$ monitoring activity for which the $\text{PM}_{2.5}$ reference method measurements are to be entered into the Aerometric Information and Retrieval System (AIRS) database. The recommendations and guidance in this document—to the extent that they go beyond the specific regulatory requirements set forth in the method or in the sampler-specific requirements contained in a particular sampler's operation or instruction manual—are not mandatory or binding in a formal sense. However, monitoring agencies are strongly encouraged to adopt and follow the recommendations and guidance to help ensure that monitoring data are of acceptable quality. Following the recommendations and guidance herein is also strongly encouraged for any $\text{PM}_{2.5}$ monitoring activity using reference or Class I equivalent methods where a high level of data quality is needed.

^{*}All references are listed in Section 14.0.

This document is not a full description of a PM_{2.5} monitoring method and does not substitute for the formal method description as set forth in the PM_{2.5} reference method (EPA 1997a) or for the sampler-specific requirements contained in the associated operation or instruction manual, which is an official part of each designated reference or equivalent method for PM_{2.5}.

1.3 Conventions

Where this document refers to mandatory method requirements, the terms “shall” and “must” are used, and a supporting reference to the applicable section of the reference method regulation is generally provided. Use of the word “should” indicates an activity or procedure that is strongly recommended to help achieve a high level of measurement data quality but is not formally required by the method itself. Finally, the word “may” is used to indicate activities or suggestions that are optional or discretionary. The use of these terms is generally consistent with American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994 guidance (ANSI/ASQC 1994).

1.4 Format and Structure

The format and structure of this document are somewhat different from the format and structure of previously published handbook documents. The page format has been updated to a new uniform standard that will be used for future sections of the *Quality Assurance Handbook*. It features a common, easily read text font and a new standard bold sans serif font for headings. New uniform header and footer information indicates the organizational position of each page in the overall *Quality Assurance Handbook* structure, giving the volume number, document number, and subject identification, the major section number and title, the section page number, and the revision date.

The organization of the document has also been modified somewhat from that used previously, with some new or changed section titles and some entirely new section topics. These changes are intended to align the structure more closely with the organizational structure and topics of SOPs recommended by EPA’s Quality Assurance Division (QAD) in its G-6 guidance (EPA 1995). This greater alignment will help monitoring agencies use this document when preparing their individual SOPs for PM_{2.5} monitoring. It should be clearly understood, however, that this document is not a ready-made, generic SOP as is, and that it must be adapted and tailored specifically to each monitoring agency’s individual policies and circumstances to become an official agency SOP.

1.5 Overview of Reference and Equivalent Methods for PM_{2.5}

A “method” for PM_{2.5} generally consists of the following:

- A PM_{2.5} sampler or analyzer designed, built, and sold by a particular manufacturer
- An operation or instruction manual provided by the instrument manufacturer that describes the proper use and operation of the sampler or analyzer
- The other operational and QA requirements necessary to obtain reliable PM_{2.5} concentration measurements.

Methods used for monitoring PM_{2.5} in SLAMS or SLAMS-related air monitoring are required to

be either reference or equivalent methods (EPA 1997c) as designated by EPA under the requirements and provisions of 40 CFR Part 53 (EPA 1997d). For most SLAMS purposes, reference and equivalent methods may be used interchangeably, and a particular method's identity as either a reference or an equivalent method is irrelevant. However, for some purposes, such as collocated auditing, a reference method may be specifically required. A current publication, *List of Designated Reference and Equivalent Methods*, identifying all methods that have been designated as reference or equivalent methods by EPA is available from any EPA Regional Office or by writing to "Department E" (MD-46), U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

1.5.1 Reference Methods

All formal sampler design and performance requirements and the operational requirements applicable to reference methods for $PM_{2.5}$ are specified in Appendix L of 40 CFR Part 50 (EPA 1997a). These requirements are quite specific and include explicit design specifications for the type of sampler, the type of filter, the sample flow rate, and the construction of the sample collecting components. However, various designs for the flow-rate control system, the filter holder, the operator interface controls, and the exterior housing are possible. Hence, various reference method samplers from different manufacturers may vary considerably in appearance and operation. Also, a reference method may have a single filter capability (single sample sampler) or a multiple filter capability (sequential sample sampler), provided no deviations are necessary in the design and construction of the sample collection components specified in the reference method regulation. A $PM_{2.5}$ method is not a reference method until it has been demonstrated to meet all the reference method regulatory requirements and has been officially designated by EPA as a reference method for $PM_{2.5}$.

1.5.2 Equivalent Methods

Equivalent methods for $PM_{2.5}$ have a much wider latitude in their design, configuration, and operating principle than reference methods. These methods are not required to be based on filter collection of $PM_{2.5}$; therefore, continuous or semicontinuous analyzers and new types of $PM_{2.5}$ measurement technologies are not precluded as possible equivalent methods. Equivalent methods are not necessarily required to meet all the requirements specified for reference methods, but they must demonstrate both **comparability** to reference method measurements and similar $PM_{2.5}$ **measurement precision**.

The requirements that some (but not all) candidate methods must meet to be designated by EPA as equivalent methods are specified in 40 CFR Part 53. To minimize the difficulty of meeting equivalent method designation requirements, three classes of equivalent methods have been established in the 40 CFR Part 53 regulations, based on a candidate method's extent of deviation from the reference method requirements. All three classes of equivalent methods are acceptable for SLAMS or SLAMS-related $PM_{2.5}$ monitoring. But not all types of equivalent methods may be equally suited to various $PM_{2.5}$ monitoring requirements or applications.

Class I equivalent methods are very similar to reference methods, with only minor deviations, and must meet nearly all of the reference method specifications and requirements. The requirements for designation as Class I equivalent methods are only slightly more extensive than the designation

requirements for reference methods. Also, because of their substantial similarity to reference methods, Class I equivalent methods operate very much the same as reference methods, and most of the information and guidance in this document will be applicable to Class I equivalent methods.

Class II equivalent methods are filter-collection-based methods that differ more substantially from the reference method requirements. The requirements for designation as Class II methods may be considerably more extensive than for reference or Class I equivalent methods, depending on the specific nature of the variance from the reference method requirements. The information and guidance in this document may be only partially applicable to Class II equivalent methods. The operation or instruction manual associated with Class II equivalent methods will, therefore, have to be more detailed in some areas than for reference or Class I equivalent methods to provide information and guidance not covered by this document.

Class III equivalent methods cover any $PM_{2.5}$ methods that cannot qualify as reference or Class I or II equivalent methods because of more profound differences from the reference method requirements. This class encompasses $PM_{2.5}$ methods such as continuous or semicontinuous $PM_{2.5}$ analyzers and potential new $PM_{2.5}$ measurement technologies. The requirements for designation as Class III methods are the most extensive, and, because of the wide variety of $PM_{2.5}$ measurement principles that could be employed for candidate Class III equivalent methods, the designation requirements are not explicitly provided in 40 CFR Part 53. For similar reasons, much of the information and guidance in this document may not be applicable to operation of Class III equivalent methods.

1.6 Limitations of $PM_{2.5}$ Reference and Class I Equivalent Methods

There are several conditions or effects that limit the degree to which a $PM_{2.5}$ reference or Class I equivalent method can precisely determine the mass concentration of particulate matter in the atmosphere. Procedures to control such effects are discussed throughout this document. One limitation is maintaining the specified ambient air flow rate through the sampling inlet and filter assembly. The flow rate determines the size of the particles that will be collected. The effects of this limitation are minimized by following sampler construction requirements and by employing procedures and checks to ensure the proper flow rate is maintained within close tolerance. Refer to Section 6.0, "Calibration Procedures," and to Section 8.0, "Field Operations," for guidance.

Another important limitation involves change in the weight of a collected sample due to mishandling, chemical reactions, and volatilization. Handling procedures, choice of filter media, humidity and temperature control of the filter and sample during collection and subsequent processing, and promptness in weighing the sample following collection all help control filter artifacts. The chemical makeup of the $PM_{2.5}$ particulate matter will vary with sampling location and source. Thus, the magnitude of $PM_{2.5}$ weight changes due to chemical and physical processes will also vary with site location.

Weight loss due to mechanical removal of particles from the filter is minimized by carefully removing the filter and its cassette from the sampler, storing the filter in a protective container during transit to the weighing laboratory, and carefully removing the filter from the cassette and neutralizing the static charge on the filter before weighing. Refer to Section 7.0, "Filter Preparation and Analysis."

The choice of an essentially neutral Teflon[®] media filter as the collecting surface minimizes the weight gain that occurs when sulfate-containing particles form by chemical reactions of sulfur dioxide gas at the surface of alkaline media such as glass fiber filters. Appendix A to this document discusses this effect and gives a procedure for measuring the alkalinity of filters. If nitric acid vapor is present at a sampling location, it can deposit on a Teflon filter and cause small weight gains in proportion to the amount of nitric acid present in the atmosphere (Lipfert 1994). This weight gain may not be controllable. Weight losses can occur due to thermal or chemical decomposition or evaporation of compounds like ammonium nitrate (NH_4NO_3), which release ammonia and nitric acid as gases. Semivolatile organic compounds (SVOCs) may be part of the sample makeup; if so, they may evaporate and cause sample weight losses. Such weight losses are minimized or standardized by maintaining the sampler temperature near ambient conditions during the sampling process, keeping the sample cool during transport to the laboratory, and promptly conditioning and weighing the sample following its receipt in the laboratory. Weight gain or loss due to absorption or desorption of water vapor on the filter or on the particulate matter is minimized by specifying low moisture pickup for manufactured filters and by conditioning the filters within specified humidity and temperature ranges, both before use and after receipt from the field. Refer to Section 7.0, "Filter Preparation and Guidance," for further guidance.

Errors in the gravimetric analysis of samples can also result from the buildup of electrostatic charge on filters during their manufacture or during sampling (Engelbrecht et al. 1980). This static buildup will interfere with the microbalance weighing, but it can be reduced or eliminated by the use of Polonium-210 (^{210}Po) antistatic strips before the weighing process begins.

2.0 Prerequisites

2.1 Introduction and Definitions

The air pollutant known as PM_{2.5} is the most recent addition to the ambient air criteria pollutants, which are required by Federal law to be measured and reported on a nationwide basis. Regulations governing its measurement were effective September 16, 1997.

The intent of this document is to provide a review of monitoring requirements and to give guidance on assuring the quality of the collection of samples, the determination of the volume of air sampled, and the gravimetric determination of the amount of PM_{2.5} collected. From these data, the concentration of PM_{2.5} particles in the ambient air can be calculated and expressed as micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) of air. This document addresses procedures that apply mainly to reference and Class I equivalent methods for sampling PM_{2.5}. Although some of the terminology associated with the PM_{2.5} sampling method may be familiar to those who have operated dichotomous samplers, an annotated listing of acronyms, abbreviations, specialized terms, and other expressions is given in Table 2-1 to familiarize the new user of PM_{2.5} samplers with key terms.

2.2 Personnel Qualifications

General aspects of personnel qualifications, training, and guidance are discussed in Volume II, Part I, Section 4, of the *Quality Assurance Handbook for Air Pollution Measurement Systems* (EPA 1997). SLAMS reporting organizations or State agencies can use information from this source and specific information about PM_{2.5} sampling and analysis to develop a training program for their employees.

The responsibility for ensuring adequate training of personnel rests with the organization's management. The Quality Assurance Coordinator of the organization should be involved as well. He or she should stress to management the need for adequate training and recommend that employees be tested or examined to evaluate the success of training and identify where further emphasis is needed.

All personnel should be familiar with general environmental laboratory procedures and field measurement techniques. Those who service the PM_{2.5} sampler in the field and operate the microbalance in the laboratory must be very conscientious and attentive to details in order to report complete and high-quality PM_{2.5} data. Persons qualified to work with PM_{2.5} operations should be able to:

- Operate the PM_{2.5} sampler
- Calibrate, audit, and troubleshoot the PM_{2.5} sampler
- Use common methods to determine temperature, pressure, and relative humidity (RH) in the field and in the laboratory
- Use a microbalance and antistatic devices
- Label, organize, and archive filters and samples in the laboratory

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- Enter data into the Aerometric Information Retrieval System (AIRS) and other databases.

Suggested means for training are as follows:

- Initially, train personnel who are already familiar with high-volume and dichotomous sampler operations as well as with weighing room techniques and requirements.
- Have personnel study the contents of this QA handbook document, the samplers' operating manuals, and the *Code of Federal Regulations* (CFR) citations.
- From these studies, develop a training document and operations checklist.
- Have personnel attend State-sponsored or regionally sponsored training workshops.
- Have personnel watch and study instructional videos that may become available.
- Have experienced operators instruct others through on-the-job training.

2.3 Health and Safety Warnings

To prevent personal injury, all employees must heed any warnings that are associated with operation of the microbalance, the PM_{2.5} sampler, and their supporting equipment and supplies. Specific health and safety warnings will generally be found at the point in the operating manual or troubleshooting guide where they are most applicable.

In general, health and safety warnings will fall into these categories:

- Electrical
- Chemical
- Equipment placement and stability.

Electrical safety considerations that would apply to the PM_{2.5} sampler include the following:

- Make all electrical connections in accordance with national codes. Always use a third wire grounding arrangement on samplers and any electrical appliances or test rigs. To minimize the possibility of electrical shock and injury, always use a grounded outlet and cord. This process will avoid the possibility of electrocution.
- Electrical supply lines to the PM_{2.5} sampler must be installed so that they remain dry under all weather conditions and are protected from exposure to extremes of light and heat, which will degrade the covering and insulation. Inspect the electrical cords and connections for signs of wear and have an electrician repair or replace them as needed.
- Always unplug the power to the sampler when servicing or replacing parts in areas requiring removal of protective panels.
- Avoid contact of jewelry with electrical circuits. Remove rings, watches, bracelets, and necklaces to prevent shorting and electrical burns.
- Use caution when working near moving parts (such as pumps) to avoid injury.
- Be aware of weather patterns; leave the area if lightning storms approach, since the sampler is elevated and made of conductive metal.

Chemical safety considerations that apply to PM_{2.5} operations include the following:

- Use care in the application of cleaning solvents, greases for O-rings, and silicone oil for the impactor well. Use of gloves is recommended. Wash hands thoroughly after working with chemicals. Provide good ventilation if organic solvents are used. Dispose of chemicals and shop towels properly.
- Mercury metal, a poisonous material, is present in some types of barometers and RH indicators. If liquid mercury is spilled, it must be cleaned up and disposed of properly. Use protective equipment to avoid inhalation of vapors and impermeable gloves to avoid skin contact. Mercury cleanup kits are available.
- Exercise caution when using antistatic devices containing radioactive polonium sources. Keep an inventory of the location and size of antistatic devices. Dispose of the devices in accordance with State and local regulations.

Equipment placement and stability are important as well. Ensure that the PM_{2.5} sampler(s) and associated equipment are stably mounted to the ground or other surface so that they do not tip over. Electrical wires should be installed so no one will trip over them and be injured. If the sampler is placed on the roof of a building or on another elevated location, ensure that railings are installed and warning signs are placed to prevent falls.

2.4 Cautions

Damage to the PM_{2.5} sampler may result if caution is not taken to properly install and maintain the device. Follow the manufacturer's instructions for maintenance of the pump, for cleaning of the interior and exterior surfaces, and for safe, secure installation.

The weighing room must be maintained in a state of good order so that samples are not contaminated, misplaced, or misidentified. The room air must be relatively dust-free and controlled within specified temperature and humidity ranges. The microbalance must be placed on a vibration-free mounting. Care must be taken to avoid bumping the balance or else its calibration settings may be disturbed. The calibration weights used to check balance precision and accuracy must be stored in a secure location away from sources of corrosion and should be used only for filter weighing, used by a knowledgeable analyst, and handled only with plastic tweezers to prevent scoring and weight changes.

Finally, care must be exercised in handling new and used filters. If details concerning weighing, labeling, and transporting of filters are not followed to the letter, errors will result. Rough handling of used filters may dislodge PM_{2.5} material. Inadequate conditioning of filters at the specified temperatures and humidities or long delays between sample retrieval and sample weighing may lead to positive or negative weight changes and thus inaccurate PM_{2.5} concentrations.

3.0 Summary

3.1 Introduction

As described in 40 CFR Part 50, Appendix L (EPA 1997a), the reference method for $PM_{2.5}$ sampling employs a sampler to draw a measured quantity of ambient air at a constant volumetric flow rate through a specially designed particle-size discrimination inlet. $PM_{2.5}$ particles are those particles with an aerodynamic diameter of less than or equal to a nominal $2.5\ \mu\text{m}$. Particles in the $2.5\ \mu\text{m}$ and smaller size range are collected on a 47-mm diameter Teflon[®] filter during the specified 23- to 25-hour sampling period. Each filter is weighed before use and after sampling. From these measurements, the mass of the collected $PM_{2.5}$ sample can be calculated.

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The concentration of $PM_{2.5}$ in the ambient air is computed as the total mass of collected particles in the $PM_{2.5}$ size range divided by the total volume of air sampled and measured under ambient (actual) conditions of temperature and pressure. The $PM_{2.5}$ concentration is thus expressed as $\mu\text{g}/\text{m}^3$ of air. The particle-size discrimination characteristics of the sampler inlet, downtube, fractionator, and filter holder are all specified by design in 40 CFR Part 50, Appendix L (EPA 1997a). In addition, specific performance characteristics of the sampler are tested in accordance with the procedures in 40 CFR Part 53, Subpart E (EPA 1997b). Sampling methods for $PM_{2.5}$ that meet all requirements in both Parts 50 and 53 are designated as $PM_{2.5}$ Federal Reference Methods (FRMs) for use in SLAMS and Prevention of Significant Deterioration (PSD) monitoring networks. Those designated methods are identified by a specific number and can also be identified by the manufacturer and model number of the sampler.

The procedures provided in this document are designed to serve as guidelines for the development of State or local agency QA programs in support of measurements of $PM_{2.5}$ by either FRM samplers or Class I equivalent method samplers.

3.2 Illustrations of Sampler Inlet Components and Sample Flow Paths

Figure 3.1 illustrates the inlet of the $PM_{2.5}$ sampler. This inlet is designed to representatively extract ambient aerosols from the surrounding airstream and remove particles with aerodynamic diameters greater than $10\ \mu\text{m}$ and to send the remaining smaller particles to the next stage. Figure 3.2

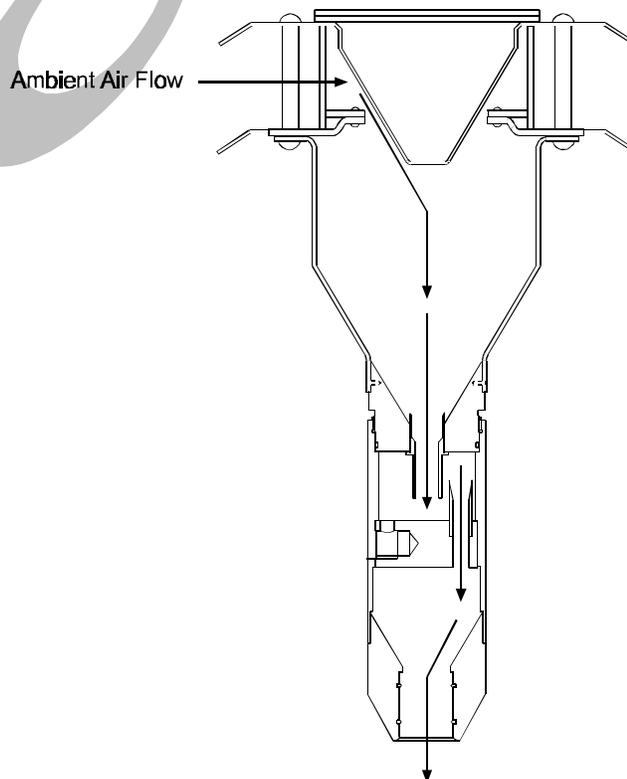


Figure 3.1. Air flow through $PM_{2.5}$ sampler inlet head.

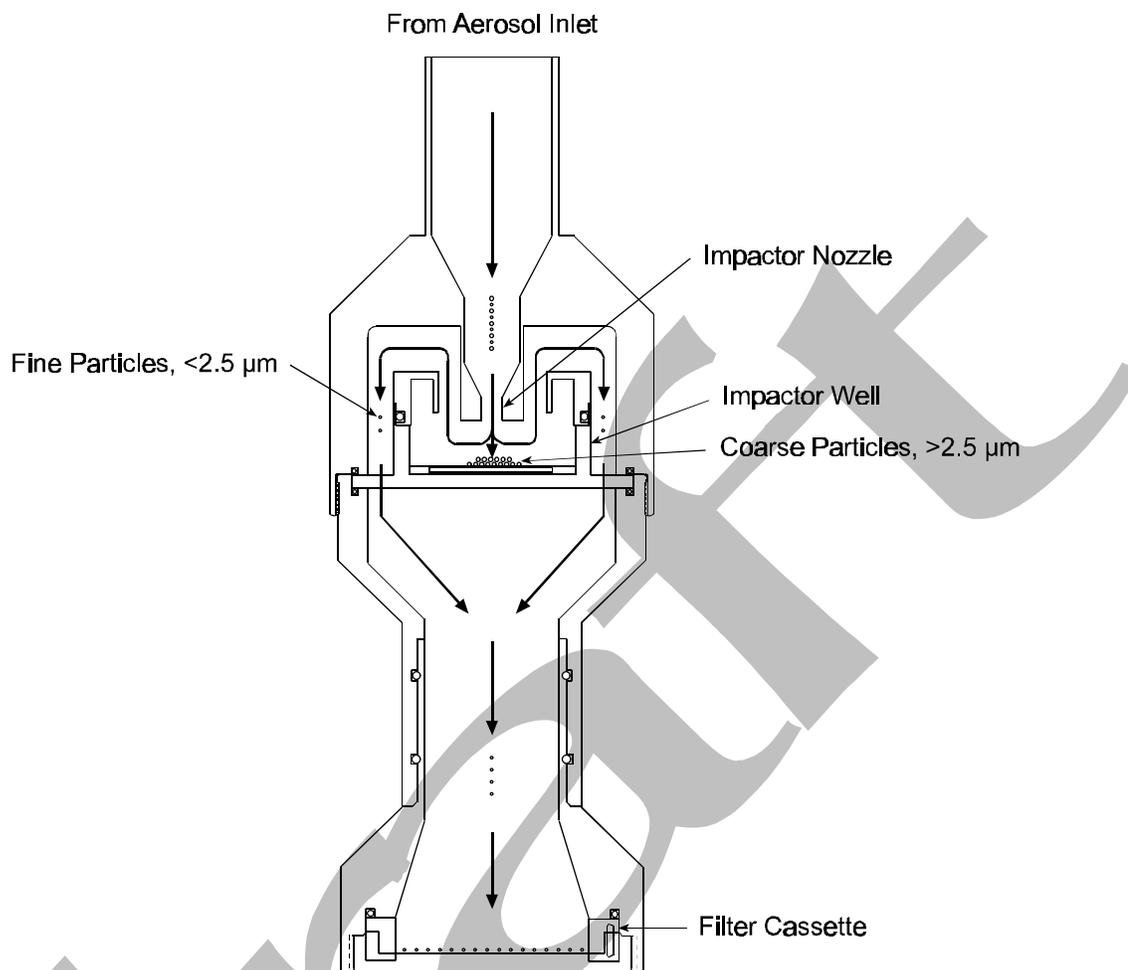


Figure 3.2. Air flow through PM_{2.5} impactor well and filter holder.

illustrates the impactor and filter holder assembly that first removes those particles less than 10 µm but greater than 2.5 µm in diameter but allows particles of 2.5 µm in diameter to pass and be collected on a Teflon filter surface. Downstream of the inlet, particles less than 10 µm but greater than 2.5 µm are removed by a single-stage, single-flow, single-jet impactor assembly. The well of the impactor assembly contains a 37-mm diameter glass fiber filter that is immersed in 1 mL of low volatility, low viscosity diffusion oil. The oiled glass fiber filter helps remove particles between 10 and 2.5 µm in diameter by preventing bouncing of the incoming particles off the loaded impactor.

3.3 Overview of Contents of This Document

The contents of this document are presented in the following sections:

- **Section 1.0**—Scope and Applicability (reference and equivalent methods; limitations)

- **Section 2.0**—Prerequisites (key word definitions, personnel qualifications, and training; health and safety warnings; cautions)
- **Section 3.0**—Summary (PM_{2.5} method and contents of document)
- **Section 4.0**—Procurement of Equipment and Supplies
- **Section 5.0**—Installation (at field site; includes notes on collocated samplers)
- **Section 6.0**—Calibration Procedures (temperature, pressure, flow rates)
- **Section 7.0**—Filter Preparation and Analysis (filter conditioning and weighing)
- **Section 8.0**—Field Operations
- **Section 9.0**—Maintenance
- **Section 10.0**—Auditing Procedures (including temperature, pressure, flow rate, and collocation of samplers)
- **Section 11.0**—Calculations, Validations, and Reporting of PM_{2.5} Monitoring Data
- **Section 12.0**—Data and Records Management
- **Section 13.0**—Assessment of Measuring Uncertainty for Monitoring Data
- **Section 14.0**—References
- **Section 15.0**—Index
- **Appendix A**—Measuring Alkalinity of Filters

3.4 Summary of Sampling Procedures for PM_{2.5}

To illustrate the steps taken to install a filter/cassette in the sampler to begin a run and the steps taken to shut down the sampler at the end of a run, an outline or checklist to follow is given in Figure 3.3.

I. Prepare for Site Visit on Scheduled Date/time

Be sure to take the following equipment and supplies to each of the sites:

- Prew weighed sampling filter in cassette, packed in a labeled metal carrier. Also take spares.
- Three preweighed field blank filters in cassettes, packed in labeled metal carriers, if a field blank study is scheduled
- PM_{2.5} Sampler Run Data Sheet for each sampler, site notebook; calculator
- Transfer standard for ambient temperature measurements
- Transfer standard for ambient atmospheric pressure measurements
- Transfer standard for volumetric flow-rate measurements
- Laptop computer and connecting cables to download sampler data
- Spare parts and tools to include O-rings, silicone grease, lab wipes, voltmeter, etc.
- Operator's manual for sampler(s) to be serviced
- If it is time for the recommended every-fifth-day checks, also take the following:
 - Clean impactor well assembly or filter/lab wipes/diffusion oil to clean and service the one at the site
 - Sample inlet adapter and flow rate measurement transfer standard
 - Clean, unused flow check filter in its cassette
 - Sampler Flow Check Data Sheet

II. Install Filter/Cassette and Begin Sampler Operations

Note: If a sampling run is ending, first follow instructions for removing sample/cassette. Refer to Part III. If the schedule calls for any of the following procedures, carry them out before installing a new sampling filter/cassette:

- Impactor well replacement or cleaning
- Sampler flow check
- Sampler calibration (temperature, pressure, flow rate)
- Cleaning of sampler inlet head and downtube
- Leak check
- Field blank study.

Carry out the following steps to install the filter/cassette and begin sampler operations:

- If sampler is programmed to begin sampling at a preset time, be sure to arrive early enough beforehand to carry out all procedures.
- Be sure sampler is **not** operating.
- Fill in initial information on PM_{2.5} Run Data Sheet.
- Remove the new filter/cassette from its protective metal case and visually inspect the filter/cassette for flaws. Verify that this is the correct filter for this sampler, site, and run date.
- Remove the sampler's filter holder assembly (if required by the manufacturer's instructions). Inspect the O-rings inside the filter holder.
- Install the filter/cassette in the filter holder assembly, and then install the loaded filter holder assembly in the sampler per the manufacturer's instructions. If you touch or scratch the filter, void the filter and get another one from the set of extra filters brought to the site.
- Program the sampler to energize at the beginning of a sampling period (consult the instruction manual).
- Make independent measurements of ambient temperature (T_a) and ambient pressure (P_a) using transfer standards. Record these values and the T_a and P_a values indicated by the sampler on the data sheet.
- If the operator is present at the time, ensure that the sampler(s) begins operation at the designated time. Record the start time on the data sheet. If the operator is present at the time, 15 minutes after sampling begins, record the sampler's display value for the indicated flow rate, Q, in L/min on the data sheet. If operator cannot be present, record the information later based on downloaded data from the run.

(continued)

Figure 3.3. Summary of sampling procedures for PM_{2.5}.

III. Remove Filter/Cassette; End Sampling Operations

Carry out the following steps to remove the filter/cassette and end sampling operations:

- If possible to do so, arrive at site before sampling period ends.
- Determine P_a and T_a using transfer standards. Enter on data sheet.
- When sampling ends, record stop time, total elapsed time, final Q , Q_{avg} , Q_{cv} , total volume sampled, T_a , P_a , etc, on data sheet.
- After each completed run, download data from the sampler data port to a laptop or other computer storage disk.
- Open the filter holder assembly (consult the instruction manual); remove the used filter/cassette; visually inspect the filter for tears, oil, insects, moisture, etc; and record observations on the data sheet.
- Place the filter/cassette inside a properly labeled protective container. Verify the container's label versus the site name, date, etc.
- Place the metal container inside a cooled storage chest. Do not allow the metal container to come into contact with ice or water. Sealed cooling blocks are recommended. Protect the containers from condensed water.
- Inspect the interior of the filter housing. Note any abnormalities.
- Inspect the interior of the impactor housing and the exterior of the impactor well. Remove any moisture or dust with a lint-free wipe and make notes on the data sheet.
- Without opening the impactor well, inspect the well's interior. Note any abnormalities. Clean or replace the impactor well if necessary or if the recommended 5-day servicing is due. Reinstall the impactor assembly. (If another sampling run is to begin, insert a new filter/cassette in the filter holder assembly and set up the sampler for the next run.)
- Review the recorded data for sample elapsed time, flow rate, filter quality, and temperature to start the process of determining if the sample is valid, questionable, or invalid. Scan through the sampling summary on the sampler display and note flags. Record observations and reasoning for questioning or invalidating a run on the data sheet.
- Make a final check of the site, and observe and record the presence of any activity that may have affected the particulate loading of the sample.
- Keep the metal container holding the filter/cassette at a temperature of less than 25 °C (preferably cooled to 4 °C), and promptly deliver it and the original of the data sheet to the sample custodian or balance operator in the weighing laboratory. Keep a copy of the data sheet with the site records.

Figure 3.3 (continued)

4.0 Procurement of Equipment and Supplies

4.1 Overview

The establishment of an ambient PM_{2.5} air monitoring network requires the procurement of specialized equipment and supplies for field operations and subsequent laboratory determination of particle mass. Information in this section will assist reporting organizations and States in selecting the proper equipment and in acceptance testing. Section 4.2 describes field operation equipment (excluding sampling filters) and Section 4.3 describes laboratory equipment (including sampling filters). Note, sampling filters are described in Section 4.3 because they are purchased for the laboratory, which must equilibrate and preweigh them before field use. In addition to field operations and laboratory equipment, data handling system(s) (including forms, logs, files, and reporting procedures) must be developed and implemented. Information on data handling systems is provided in Section 12.0, "Data and Records Management."

It is recommended that minimum monitoring equipment requirements and budgetary limits be established before equipment is purchased. In addition, acceptance criteria for equipment and supplies should be established. Upon receipt of the sampling equipment and supplies, these acceptance checks should be conducted. The results of these checks, including whether the equipment was accepted or rejected, should be recorded in a procurement log. Figure 4.1 is an example of such a log. This log will serve as a permanent record for procurement and provide cost projections for future programs. Table 4-1 lists the major equipment needed, how it should be tested, suggested acceptance limits, and actions to be taken if acceptance limits are not met.

It is impossible to include every item that might be needed by any specific monitoring network. Therefore, each agency must determine the extent of its in-house inventory and the items that should be ordered before sampling can begin. The agency must also be prepared to order any additional equipment required over and above that outlined in this section.

Item	Description	Qty	PO #	Vendor	Date		Cost	Initials	Accept/ Reject	Comments
					Ord.	Rec'd				
1 case filters	2 µm Pore 47 mm diameter	60	971- 100	WIZ Suppl y	8/1/97	8/15/9 7	\$100	ABC	Accept	None

Figure 4.1. Example procurement log.

TABLE 4-1. ACCEPTANCE TEST AND LIMITS FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance check	Acceptance limits	Action if requirements are not met
Field operations			
Sampler	Sampler complete; no evidence of damage. Model designated as reference or equivalent method	Specifications outlined in 40 CFR Part 50, Appendix L	Reject sampler
Calibration QA/QC equipment for flow rate, temperature, pressure, etc.	Accompanied by certificate. Check values against National Institute of Standards and Technology (NIST)-traceable standards	Within accuracy limits described in this document	Adjust or reject equipment
Laboratory operations			
Filters	Of correct type and undamaged	Type as described in 40 CFR Part 50, Appendix L	Reject filters
Filter cassettes	Of correct type and undamaged	As specified by sampler manufacturer	Reject filter cassettes
Filter/cassette protective containers	Of correct type and undamaged	As described in this document	Reject protective containers
Filter-handling containers	Of correct type and undamaged	As described in this document	Reject filter-handling containers
Analytical microbalance	Accompanied by certificate. Check values against NIST-traceable standards	Within accuracy limits described in this document	Adjust or reject equipment
Calibration weights	Accompanied by certificate. Check weights against NIST-traceable standards	Within accuracy limits described on certificate	Reject weights

4.2 Procurement Prerequisites—Field Operations

4.2.1 Reference or Equivalent Method Sampler

Each PM_{2.5} sampler used for reporting data to determine attainment of the National Ambient Air Quality Standard (NAAQS) for fine (2.5 μm) particulate matter **must** meet U.S. EPA standards and **must** be of a model designated by EPA as a reference or equivalent method. The minimum sampler requirements are described in the reference method (EPA 1997a) and in 40 CFR Part 53 (EPA 1997b). The PM_{2.5} sampler cost will vary with the manufacturer and the sophistication of the sampler. Basic considerations for sampler selection include flow control and measurement

systems, maintenance requirements, reliability, ease of operation, and such additional capabilities as sequential sampling.

Although a network may decide to use any number of different reference and designated equivalent PM_{2.5} monitor models, using a single model of sampler in a network minimizes the variety of spare parts required to keep the network in operation. An in-house inventory of general maintenance supplies and replacement parts is recommended. Examples include: various hand tools, laboratory wipes, soft brushes, and cotton swabs. Spare parts for the sampler may be obtained from the manufacturer or may be purchased from other suppliers.

The impactor requires a circular, 35- to 37-mm diameter filter made of borosilicate glass with no binder. The filter pore size must be 1 to 1.5 μm and the thickness must be 300 to 500 μm . The impactor oil is tetramethyltetraphenyl-trisiloxane, single-compound diffusion oil, with the following specifications at 25 °C: vapor pressure, 2×10^{-8} mm Hg; viscosity, 36 to 40 centistokes; density, 1.06 to 1.07 g/cm³.

4.2.2 Calibration Equipment

Calibration activities require specialized equipment to calibrate the sampler for temperature, barometric pressure, and volumetric flow measurements. At a minimum, the following equipment is required:

- A thermometer capable of accurately measuring ambient temperatures, in the range from -30 to +45 °C, to the nearest 0.1 °C. This thermometer should be referenced (EPA 1995; NIST 1986, 1988, 1989) to within an accuracy of ± 0.1 °C to a NIST-certified thermometer or an American Society for Testing and Materials (ASTM) thermometer. Multiple thermometers may be used to cover the temperature range as long as each thermometer meets the accuracy and readability requirements described above. Thermistor or thermocouple thermometers for use in the field are calibrated against the traceable laboratory mercury thermometers.
- A barometer capable of accurately measuring barometric pressure (ASTM 1995) over a range of 600 to 800 mm Hg (80 to 106 kPa) to the nearest millimeter of Hg. At least once a year, this barometer should be referenced to within ± 5 mm Hg of a NIST-traceable standard of known accuracy. A portable, aneroid barometer (e.g., a climber's or engineer's altimeter) is suitable for field use. A Fortin-type, mercury-column barometer may also be useful for laboratory checks of the barometer used for field measurements.
- Flow-rate measurement equipment (transfer standards) capable of accurately measuring the volumetric flow rate at the temperature and pressure of the sample air entering the PM_{2.5} sampler. A variety of flow-rate transfer standards, with their optimum flow ranges and their support equipment are presented in Section 6.3.2. The flow-rate transfer standard's calibration must be referenced annually to within ± 2 percent of a NIST-traceable primary standard.
- A flow-rate measurement adapter of the dimensions specified in drawing no. L-30 in Appendix L of 40 CFR Part 50 (EPA 1997a) and described in Section 7.3.6, Appendix L, that will connect the transfer standard outlet to the PM_{2.5} sampler flow path entering the impactor assembly and form a leak-free seal. All interconnecting tubing should be flexible and crimp-resistant. This adapter is also used in conducting the required external leak test.

4.2.3 QC Flow-Check Device

As part of QC procedures, a flow-check device is required to verify that the $PM_{2.5}$ sampler is operating at the correct flow rate. Figure 4.2 shows construction details for an inexpensive and rugged QC flow-rate check device that is based on measurement of the pressure drop across an orifice. This device is intended for use with a water manometer or other differential pressure meter that will read in inches of water. Note that proper calibration of the orifice must be conducted in order to accurately predict the device's response to variations in ambient temperature and pressure. Orifices may be calibrated versus a NIST-traceable soap-bubble flowmeter (Nelson 1971). Other calibrated volumetric flow-rate devices such as the flow-rate transfer standards described in Section 6.3.2 may also be used to check flow rates during routine operation.

4.2.4 Audit Equipment

Audit equipment will be similar to the calibration equipment, described above. However, all audit equipment must be separate from the equipment used for calibration and flow checks, although they may be of identical makes and models.

4.3 Procurement Prerequisites—Laboratory Operations

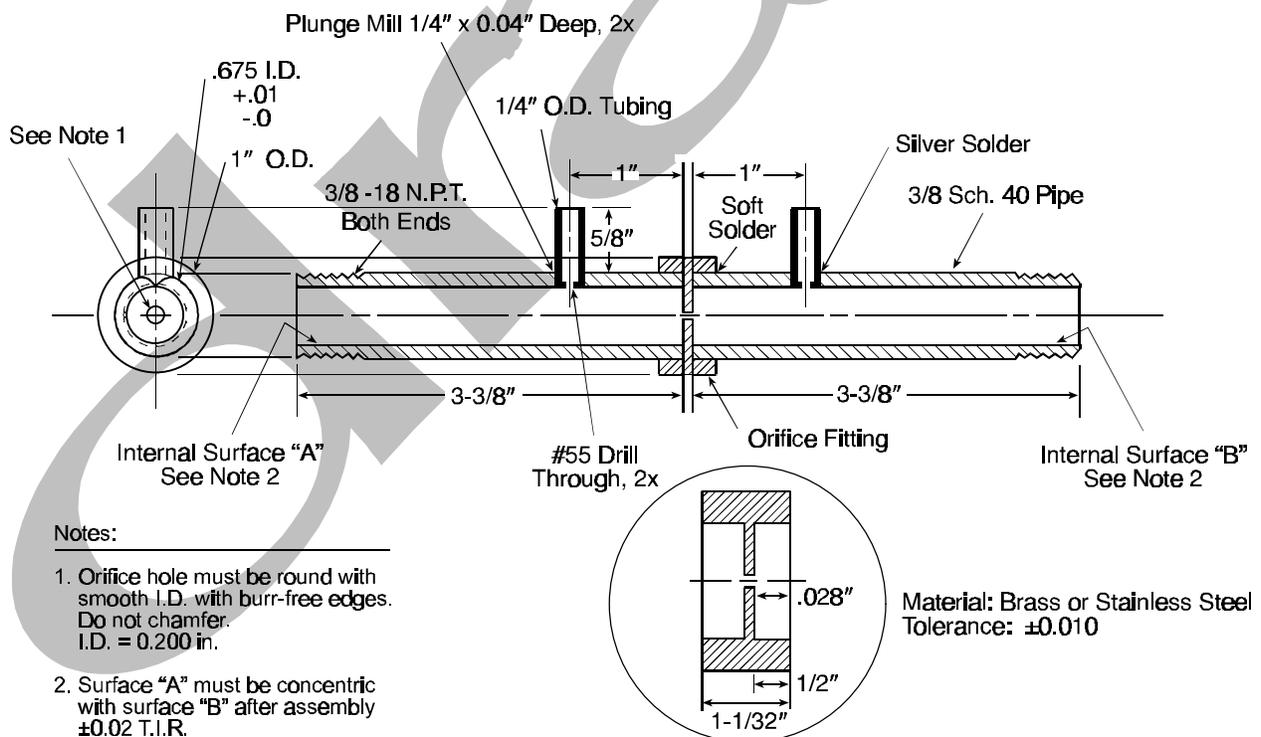


Figure 4.2. Orifice dimensions to provide approximately 1 in. of water pressure drop at 16.7 L/min (all dimensions in inches).

4.3.1 Filter Media

In the laboratory, sampler filters are conditioned (while in a glass or plastic filter-handling container such as a petri dish), preweighed, and placed first into sample filter cassettes and then into a protective metal container for transport to the field. The mean relative humidity in the laboratory and/or the conditioning chamber should be held between 30 and 40 percent, with a variability of not more than ± 5 percent over 24 hours. Mean laboratory temperature should be held between 20 and 23 °C, with a variability of not more than ± 2 °C over 24 hours. The time required to condition filters (Section 7.6) may be as long as 6 weeks. Adequate numbers of filters must be purchased to permit conditioning before use. In addition, it is recommended that a sufficient inventory of more than one production lot of filters be maintained (in case problems are found with a particular lot). In practice, this suggestion means that networks should plan on keeping a minimum filter inventory sufficient for 6 to 12 weeks of operational needs. These inventory levels can be adjusted based on the network's experience with conditioning times for a given vendor's filters.

Only sampling filter media approved by EPA for use with PM_{2.5} samplers should be purchased. The manufacturer must certify that these media meet the specifications given in Appendix L of 40 CFR Part 50 (EPA 1997a), which are summarized below:

- Size—circular, 46.2-mm diameter ± 0.25 mm (with support ring)
- Medium—polytetrafluoroethylene (PTFE) Teflon with integral support ring
- Support ring—polymethylpentene (PMP) or equivalent inert material, 0.38 ± 0.04 mm thickness, outer diameter 46.2 ± 0.25 mm, and width of 3.68 mm.
- Pore size—2 μm as measured by ASTM F 316-94
- Thickness—30–50 μm
- Maximum pressure drop (clean filter)—30 cm H₂O column at 16.67 L/min clean air flow
- Maximum moisture pickup—No more than 10 μg weight increase after 24-hour exposure to air at 40 percent RH, relative to the weight after 24-hour exposure to air at 35 percent RH
- Collection efficiency—greater than 99.7 percent, as measured by the dioctylphthalate (DOP) test (ASTM D2986-91) with 0.3 μm particles at the sampler's operating face velocity
- Filter weight stability—filter weight loss ≤ 20 μg , measured as specified in 40 CFR Part 50, Appendix L
- Loose surface particle contamination— < 20 μg as determined by procedure in 40 CFR Part 50, Appendix L
- Temperature stability—weight change after heating to 40 ± 2 °C must be less than 20 μg as determined by procedure in 40 CFR Part 50, Appendix L
- Alkalinity—less than 25 microequivalents/g of filter, as measured in the procedure given in Appendix A to this document.

Although not required for determination of PM_{2.5} mass concentration under this reference method, additional specifications for the filter must be developed by users who intend to subject archived

PM_{2.5} filter samples to subsequent chemical analysis. These supplemental specifications include background chemical contamination of the filter and any other filter parameters that may be required by the chemical analysis method. All such supplemental filter specifications must be compatible with and secondary to the primary filter specifications given in Appendix L of 40 CFR Part 50 (EPA 1997a).

4.3.2 Filter Support Cassettes

Most PM_{2.5} samplers use filter support cassettes (see Figure 7.3 and 40 CFR Part 50, Appendix L, Figures L-27 through L-29) to hold the sampling filter while in use. The filters are placed into these cassettes in the laboratory. The cassettes, with the filters in place, are then transported to the field in protective containers (see Section 4.3.3). A sufficient number of cassettes must be purchased to allow all placement and removal of filters in cassettes to be performed in the laboratory. Under no circumstances should filters be placed in or removed from the filter support cassettes in the field.

4.3.3 Filter Cassette Protective Containers

Filter cassettes should be transported between the laboratory and the field in protective metal containers, as illustrated in Figure 7.3. Suitable containers should be available from the manufacturer or from a supplier specified by the manufacturer. Containers should be designed to prevent the cassette from moving within the container during transport. To avoid losses of particulate matter, do not allow the surface of the filter to touch any interior surfaces of the protective container. Containers should be marked for identification purposes. Plastic protective containers have been found to be unacceptable due to their potential for buildup of static electrical charges which can cause particles to be lost from or deposited on the filter surface. Special, sampler-specific protective containers are needed for the trays or cartridges that hold multiple filters for use in sequential samplers. Consult the sampler manufacturer for details and availability.

4.3.4 Insulated Shipping Container

An insulated shipping container, capable of maintaining a temperature less than 25 °C, is needed to transport the filter cassette containers between the field and laboratory after sampling. Plastic coolers, such as those used by campers and fishers, filled with leak-proof ice substitutes have proven suitable for this use. The sample cassette temperature should be monitored from the time of collection until the sample is equilibrated (i.e., during shipping and handling). This can be done with an inexpensive electronic max-min thermometer placed in the shipping container.

4.3.5 Filter Handling Containers

Filter media (particularly the 47-mm [1.85-in.] diameter, 2- μ m pore size filter specified for use with reference or Class I equivalent samplers) are especially delicate and easily damaged. Post-sampling particle loss and filter damage may occur if proper handling procedures are not followed.

To ensure the integrity of the sample, some type of protective covering is required for filter handling within the laboratory. A glass or plastic petri dish, designed to hold these filters, is recommended. These are available from some filter manufacturers and laboratory supply houses.

The dish should be of suitable size (i.e., large enough to allow easy removal of the filter, yet small enough to prevent excessive movement within the petri dish) and should have a tight-fitting lid to prevent intrusion of dust or loss of particles during handling at the analytical laboratory. A label is affixed to the dish to provide proper documentation. A sufficient number of containers must be available to provide storage of filters during laboratory operations.

4.3.6 Analytical Microbalance

An analytical microbalance is required to weigh the sample filters. Its capacity must be adequate to weigh the sample filters (typically 100 to 200 mg). It must have sufficient room to weigh the size of filters used (i.e., 47-mm diameter). The microbalance must have minimum readability of ± 1 μg and a reproducibility of 1 μg . Readability is the smallest difference between two measured values that can be displayed by the microbalance. Repeatability is a measure of the ability of a microbalance to display the same result in repetitive measurements of the same weight under the same measurement conditions. The balance must be calibrated at installation and recalibrated immediately prior to each weighing session. ^{210}Po antistatic strips will be needed to neutralize electrostatic charge buildup in the microbalance's weighing chamber and on individual filters. Refer to Section 7.7.

4.3.7 Mass Reference Standards

Mass reference standards (or balance check weights) are required to verify proper microbalance operation as part of the normal QC operations. These standards must be ASTM Class 1, 1.1, or 2 standards traceable to NIST with a tolerance of no more than 0.025 mg. The mass reference standards should be selected so as to bracket the maximum and minimum expected filter weights. Mass reference standards should be recalibrated on a regular basis (e.g., yearly) at a NIST-accredited State weight and measures laboratory or at a calibration laboratory that is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST. The recalibration frequency should be determined from records of previous recalibrations of these standards. See Table 4-2 for specifications for a 200-mg mass reference standard.

TABLE 4-2. SPECIFICATIONS FOR 200-mg MASS REFERENCE STANDARD

ASTM Class	Individual weight tolerance (mg)	Approximate cost (\$) ^a
1	0.010	42
1.1	0.005	—
2	0.025	23

^a Costs may be \$30 (or more) higher for individual weights with a National Voluntary Laboratory Accreditation Program (NVLAP) traceable certificate or a NVLAP weight calibration report. Weights that include these certificates are traceable to NIST.

5.0 Installation

5.1 Siting Requirements

As with any type of air monitoring study in which the sample data are used to draw conclusions about a geographic area, the validity of those conclusions depends on the representativeness of the sampling data. Therefore, an initial goal of a PM_{2.5} monitoring project is to select a site where the PM_{2.5} measurements will be representative of the monitoring area.

Spatial and temporal scale considerations are important in PM_{2.5} sampler siting. Spatial scales may range from a small (0.1- to 0.5-km²) area to large regional areas exceeding tens of hundreds of square kilometers. Whether the potential impact of particulate pollution is generated by a local or a general source category will affect the decision on the size of the spatial monitoring scale. In addition, the siting of the samplers within a monitoring network should reflect whether the expected impact will be limited to a small area (a few city blocks) or will extend to larger areas (metropolitan or rural). With regard to the temporal scale, interest focuses on either an annual geometric mean concentration or a 24-hour average concentration. Because siting of a PM_{2.5} sampler requires considering the prevailing wind direction, a sampler sited for monitoring trends in air quality over a period of a year will not necessarily be ideal for measuring 24-hour concentrations. Thus, the choice of temporal scale will also affect the sampler location. These spatial and temporal aspects of network design and optimum site exposure are more completely explained in 40 CFR Part 58, Appendix D (EPA 1997a) and in a siting guidelines document to be published by the EPA Office of Air Quality Planning and Standards (EPA 1997b).

Although spatial and temporal scales must be considered in site selection, the following guidelines should be observed regardless of the scale:

- The PM_{2.5} sampler must have unobstructed air flow for a minimum of 2 m in all directions.
- The sampler inlet should be placed at a height of 2 to 15 m above ground level.
- If a PM_{2.5} sampler is collocated with any other particulate matter sampler, the spacing between sampler inlets must be at least 2 m and no more than 4 m. The heights of the inlets should be within 1 m as measured in the vertical direction.

Additional factors must be considered in determining where the sampler itself will be located. These factors include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring equipment.

A PM_{2.5} sampler used for routine sampling must be situated where the operator can reach it safely regardless of weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is not jeopardized by a slippery roof surface during inclement weather. Considerations also should be given to the fact that routine operation (i.e., calibrations, sample filter installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

In 40 CFR Part 50 Appendix L (EPA 1997c), a PM_{2.5} sampler is required to operate at 105-125 volts, AC and a frequency of 59-61 Hz. The sampler may pull a higher current when the pump starts, possibly necessitating a slow-blow fuse. Although PM_{2.5} samplers are required to indicate

power interruptions, every effort should be made to provide a stable power source for the monitoring site.

The security of the sampler itself depends largely on its location. Rooftop sites with locked access and ground level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

5.2 Sampler Installation Procedures

5.2.1. Receipt of Sampler

- On receipt of a PM_{2.5} sampler from the manufacturer, visually inspect the sampler to ensure that all components are accounted for.
- Compare the equipment delivered with the items listed on the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.
- Read the manufacturer's instruction manual and become familiar with the sampler's operating and calibration procedures.
- Assemble the sampler in the laboratory according to the manufacturer's instructions, except do not put oil in the impactor well.

5.2.2. Laboratory Evaluation

- Energize the sampler.
- Perform a leak check according to the manufacturer's instructions. Each sampler's procedure will be somewhat different from the others. The impactor should not have oil in it, and a flow check filter should be in the filter cassette for this test.
- Calibrate the flow rate at 16.7 L/min according to the manufacturer's instructions and Section 6.0, "Calibration Procedures."
- Perform audits of the temperature and pressure sensors. Refer to Section 10.0, "Auditing Procedures."
- Perform a flow check according to the manufacturer's instructions and Section 8.3.2.

5.2.3. Setup at Sampling Site

- Carefully transport the sampler to the sampling site.
- Secure the sampler in its predetermined location. The legs of the sampler can be bolted to angle aluminum bars. The bars can be held in the soil with metal stakes or they can be nailed to wooden beams if the sampler is on the surface of a roof. Keep the sampler level.

5.2.4. Field Evaluation

- Check all tubing for crimps, cracks, or breaks.

- Plug the power cord into a line voltage outlet. The use of water-proof interlocking electrical connectors is recommended to ensure operator safety and to avoid shorts and/or power interruptions. Do not allow any electrical connections to become submerged during periods of inclement weather.
- Perform another leak test with no oil in the impactor and a dummy filter in the filter cassette.
- Perform audits of the temperature and pressure sensors.
- Perform a flow check according to the manufacturer's instructions and Section 8.3.2.
- Correct any problems before proceeding.
- Install a preloaded impactor well in the sampler. The sampler is now ready for routine use.

6.0 Calibration Procedures

6.1 Overview

Before a PM_{2.5} monitoring program is undertaken, all sampling and analysis equipment must be properly calibrated. Careful, accurate calibrations of sampling instrumentation and associated equipment provide the backbone for any monitoring network. The requirements specified in this section may serve as initial acceptance checks; further checks may be necessary if acceptance specifications are not met. All required data and calculations should be recorded in a calibration logbook or on calibration data sheets. A separate section of the logbook should be used for each apparatus and sampler in the program.

According to 40 CFR Part 50, Appendix L (Subsection 10) (EPA 1997), the PM_{2.5} sampler flow-rate measurement system must be calibrated in terms of the actual volumetric flow rate at prevailing ambient conditions of temperature and pressure (Q_a), as opposed to the standard volumetric flow rate (Q_{std}), which is measured or corrected to EPA-standard conditions of temperature and pressure. Measured PM_{2.5} concentrations are also reported in terms of the actual volume at ambient conditions of temperature and pressure.* Therefore, no conversions between Q_a and Q_{std} are generally necessary. If a flow-rate standard or transfer standard has been calibrated in terms of Q_{std} , conversion to Q_a will be required. The calculations necessary to convert these flow rates are presented in Section 6.2, Equations (6-1) and (6-2).

This section presents the following aspects of calibration procedures:

- A discussion of Q_a and Q_{std} flow-rate designations and their applicability in a PM_{2.5} monitoring program (Section 6.2)
- Basic calibration procedures and associated calculations for calibration of the sampler's flow-rate measurement system (Section 6.3)
- Basic calibration procedures for the sampler's temperature and pressure measurement systems (Sections 6.4 and 6.5)
- Discussion of flow-rate transfer standards and calibration equipment (Sections 6.3.2 and 6.3.3)
- Sampler calibration frequency requirements (Section 6.6).

More detailed information regarding calibration and operation procedures can be found in the operation or instruction manual associated with each sampler.

*Note that this way of reporting PM_{2.5} measurements is different from the way PM₁₀ measurements have been reported in the past, which required that the sample volume be "corrected" to EPA-standard conditions of 25 °C and 760 mm Hg.

6.2 General Aspects of Flow-rate Measurement and PM_{2.5} Sampler Calibration

As discussed in Section 3.0, "Summary," a PM_{2.5} sampler includes a specially designed sample air inlet, a size-fractionating impactor, and a sample flow-rate control system. The particle size discrimination characteristics of both the inlet and the impactor critically depend on specific internal air velocities; a change in velocity will result in a change in the nominal particle size collected. These velocities are determined by the actual volumetric flow rate (not the mass flow rate) through each device. Therefore, the actual volumetric flow rate through the sampler's inlet and fractionator must be maintained at a constant value that is as close as possible to the design flow rate specified for the sampler. The design flow rate for a given sampler is specified in the sampler's operation or instruction manual. Upper and lower tolerance limits for the sampler flow rate are specified in 40 CFR Part 50, Appendix L.

As mentioned above, both the calibration of the sampler's flow-rate measurement system and the reporting of PM_{2.5} concentration measurements must be carried out in terms of actual volumetric units (Q_a or V_a). However, previous measurements for PM₁₀ were based on standard units (Q_{std} or V_{std}), and the certification for some calibration standards may be in terms of standard volumetric flow-rate units (Q_{std}). Therefore, it is important to understand the definition for each of these two types of volumetric units and the distinction between them. Before calibration procedures are initiated, the operating agency personnel should review the following flow-rate definitions:

- Q_a —Actual volumetric air flow rates that are measured and expressed at existing conditions of temperature and pressure are denoted by Q_a (Q_{actual}). Typical units are L/min or aL/min and m³/min or am³/min where the "a" stands for "actual." Inlet design flow rates are always given in actual volumetric flow units.
- Q_{std} —Air flow rates that have been adjusted to EPA-standard conditions of temperature and pressure (25 °C or 298 K and 760 mm Hg or 101 kPa) are denoted by Q_{std} ($Q_{standard}$). Typical units are stdL/min and stdm³/min. Standard volume flow rates are often used by engineers and scientists because they are equivalent to mass flow-rate units. Prior to 1997, standard volumes (derived from standard volume flow rates and the total time of sampling) were also required in the calculation of mass concentration ($\mu\text{g}/\text{stdm}^3$) in reporting PM₁₀ measurements.

These Q_a and Q_{std} measurement units must not be confused or interchanged. The flow-rate units can be converted as follows, provided the temperature and pressure are known. These formulas can also be used for average flow rates (\overline{Q}_a and \overline{Q}_{std}) over a sampling period by substituting average temperature (\overline{T}_a) and pressure (\overline{P}_a) over the sampling period.

$$Q_{std} = Q_a(P_a/P_{std})(T_{std}/T_a) \quad (6-1)$$

$$Q_a = Q_{std}(P_{std}/P_a)(T_a/T_{std}) \quad (6-2)$$

where

Q_{std} = standard volume flow rate, stdm³/min

Q_a = actual volume flow rate, actual m³/min

P_a = ambient barometric pressure, mm Hg (or kPa)

P_{std} = EPA standard barometric pressure, 760 mm Hg (or 101 kPa)

T_{std} = EPA standard temperature, 298 K (25 °C + 273)

T_a = ambient temperature, K (°C + 273).

Any gas flow rate measured or expressed in actual volumetric units (Q_a) is always associated with a particular temperature and pressure of the gas. If either the temperature or the pressure changes, the volumetric flow rate will also change, even though the mass flow rate of the gas remains constant. Therefore, when the flow rate is measured at different points in the sampler, the volumetric flow rate observed will be different if either the temperature or the pressure is different. For example, when a flow calibration device is connected to the sampler inlet, the pressure of the air flow measured by the flow calibration standard will be the ambient barometric pressure. The pressure of the air flow measured by the sampler's flow measurement system, however, will be somewhat lower than the ambient barometric pressure because all flow calibration devices cause some pressure drop. If this pressure drop is negligible, then there is no problem; the pressure can be considered the same for both measurement systems. But if the pressure drop is significant, then the volumetric flow rate measured by the two systems will be different, and this difference should be taken into account when comparing them. **Sampler flow-rate measurement systems may or may not automatically correct for this pressure change.** If not, one of the flow-rate measurements must be corrected to the same pressure as the other flow rate before they can be compared. This correction is easily accomplished using the following formula:

$$Q_1 = Q_2 (P_2/P_1)(T_1/T_2) \quad (6-3)$$

where

Q_1 = actual volume flow rate, am^3/min , at pressure and temperature of first point, am^3/min

Q_2 = actual volume flow rate, am^3/min , at second point, am^3/min

P_1 = pressure at first point, mm Hg (or kPa)

P_2 = pressure at second point, mm Hg (or kPa)

T_1 = temperature at first point, K ($^{\circ}\text{C} + 273$)

T_2 = temperature at second point, K ($^{\circ}\text{C} + 273$).

Note that in many cases the temperatures will be identical, so that $T_1=T_2$ and the (T_1/T_2) term will equal 1 and therefore will drop out of Equation (6-3).

6.3 Calibration of the Sampler Flow-Rate Measurement System

A full, detailed, EPA-approved calibration procedure, tailored specifically for each commercially available $\text{PM}_{2.5}$ sampler, is contained in the operation or instruction manual associated with each sampler designated as a reference or equivalent method under 40 CFR Part 53. That specific procedure should be followed carefully and thoroughly to calibrate the sampler.

CAUTION

Do not calibrate the flow-rate measurement system if there is any doubt that the temperature and pressure measurement systems are not also in calibration. If there is doubt, calibrate the T and P sensors before calibrating the flow-rate measurement system. Also check for internal and external leaks before proceeding.

This section provides additional precautions, guidance, and information on flow-rate standards and calibration that may not be contained in the specific instruction manual procedure. It also presents a generic outline of the general procedure used to calibrate the flow-rate measurement systems of commercially available samplers. This information can be used to augment the sampler-specific procedures.

6.3.1 General Requirements and Guidance

1. Multipoint calibration and single-point verification of each sampler's flow rate must be performed periodically (see Section 6.6 for frequency) to establish traceability of subsequent flow measurements to an authoritative flow-rate standard. Calibration with a flow-rate standard (or transfer standard) that is certified against a NIST-traceable standard (see Section 6.3.3 regarding NIST traceability) transfers the NIST traceability to the sampler's flow-rate measurement system. This NIST traceability is required by Appendix A of 40 CFR Part 58.
2. PM_{2.5} samplers may employ various types of flow-rate measurement devices. The specific calibration standard and procedure used for calibration or verification of the sampler's flow-rate measurement device will vary depending on the type of flow-rate measurement system employed (40 CFR Part 50, Appendix L, Sec. 9.2.1). The sampler's operation or instruction manual identifies one or more type of flow-rate or volume standard recommended for calibration and provides a detailed calibration procedure.
3. Calibration of the sampler's flow-rate measurement system must be in units of the actual ambient volumetric flow rate (Q_a) (40 CFR Part 50, Appendix L, Sec. 9.2.1).
4. The sampler flow-rate measurement system must be calibrated or verified by removing the sampler inlet and connecting the flow-rate standard to the sampler, in accordance with the instruction manual, so that the flow-rate standard accurately measures the sampler's flow rate. The operator should verify that no leaks exist between the flow-rate standard and the sampler (40 CFR Part 50, Appendix L, Sec. 9.2.3).
5. The calibration relationship between the flow rate (in actual L/min) indicated by the flow-rate standard and by the sampler's flow-rate measurement device must be established or verified in accordance with the sampler operation or instruction manual. Temperature and pressure corrections may be required for some types of flow-rate standards (40 CFR Part 50, Appendix L, Sec. 9.2.4). Consult the operations manual for the particular flow-rate standard for guidance on the nature and calculation of any corrections that may be required for the standard.
6. Calibration of the sampler's flow-rate measurement system must consist of at least three separate flow-rate measurements (a multipoint calibration) approximately evenly spaced within the range of -10 percent to +10 percent of the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). The sampler is required to have the capability to adjust the flow rate over the -10 percent to +10 percent range (40 CFR Part 50, Appendix L, Sec. 7.4.2). The sampler's instruction manual will provide additional guidance on this flow-rate adjustment.

7. Verification of the sampler's flow rate shall consist of one flow-rate measurement at the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). This one-point verification of the flow-rate measurement system may be substituted for a three-point calibration, provided that a full three-point calibration is carried out upon initial installation of the sampler and at least once per year and the flow-rate measurement system has met the ± 2 percent accuracy requirement in the previous three-point calibration. A full three-point calibration must be carried out whenever a one-point verification indicates that the sampler's flow-rate measurement system differs by ± 4 percent or more from the flow rate measured by the flow-rate standard, and the one-point verification must be repeated after the three-point calibration (40 CFR Part 50, Appendix L, Sec. 9.2.5).
8. Following a calibration or verification, the flow-rate standard is removed or disconnected from the sampler, and the sampler's inlet is carefully reinstalled. Then the sampler's normal operating flow rate must be determined (in aL/min) with a clean filter in place, using the sampler's flow-rate measurement system. If the sampler flow rate differs by ± 2 percent or more from the required sampler flow rate, the sampler flow rate must be adjusted to the specified flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.6).

6.3.2 Flow-Rate Calibration Standards

A flow-rate standard must be used for calibrating or verifying the sampler's flow-rate measurement device within an accuracy of ± 2 percent. This flow-rate standard must be a separate, stand-alone device designed to connect to the flow-rate measurement adapter. The flow-rate standard must have its own certification and must be traceable to a NIST primary standard for volume or flow rate (40 CFR Part 50, Appendix L, Sec. 9.1.2). See Section 6.3.3 below for more information on NIST traceability.

All flow-rate calibration standards must be fully equilibrated to the temperature of the air whose flow rate is to be measured. This temperature equilibration can take up to an hour, depending on the temperature at which the instrument was stored prior to moving it to the point of use. Consequently, the standard should be moved to the point of use well before it is used for calibration. During this thermal equilibration period, the standard must be exposed to the prevailing air temperature, but it also must be protected from precipitation, wind, dust, solar heating, and other hazards that could affect its accuracy.

Various types of flow-rate standards that might be considered for sampler calibration follow:

1. **Bubble flowmeters**—Although completely manual bubble flowmeters are available, a semi-automatic instrument such as the Gilian* flowmeter is easier to use and is more likely to provide more uniform flow-rate measurements. Because the liquid used to form the bubbles is usually water-based, a correction may be required to compensate for a volume increase due to evaporation of the water in the instrument, and the instrument cannot be used below 0 °C because of freezing.

*Mention of commercial products should not be interpreted as endorsement. This product is given as a typical and perhaps well-known example of the general class of instruments; other instruments in the class are available and may be fully acceptable.

2. **Piston flowmeters**—Automatic dry-piston flowmeters such as the BIOS* flowmeter are relatively easy to use and not subject to the water vapor correction or the liquid freezing limitation of the bubble flowmeters. However, they may still be subject to a similar lower operational temperature limit.
3. **Mass flowmeters**—Although mass flowmeters may be considered as calibration standards, they have a number of shortcomings in this application. They have a high pressure drop, which could change the sampler flow rate or otherwise affect the operation of the sampler. Mass flowmeters also require conversion of the mass flow measurement to the actual volumetric flow rate. The flow rate measured by the sampler may also have to be corrected to account for significant pressure drop introduced by the device itself. Finally, mass flowmeters are subject to zero or span drift, particularly at the wide range of temperatures encountered at field sites, so their accuracy should be reverified frequently over a wide temperature range.
4. **Orifice devices**—Orifices are simple and reliable, but they too require corrections for temperature and pressure and may have a very significant pressure drop. They also require a very accurate and sensitive differential pressure measurement device such as a manometer or alternative aneroid differential pressure gauge, whose accuracy must be reverified frequently. Electronic micromanometers are convenient to use with an orifice device.
5. **Laminar flow elements**—Laminar flow elements generally have a lower pressure drop than orifice devices but otherwise have the same requirements and disadvantages.
6. **Wet test meters**—Wet test meters are generally not practical for field use but may be used for laboratory calibrations.
7. **Dry gas meters**—Dry gas meters may be considered for field calibration. Some meters may have a substantial pressure drop. They should be checked for leaks and mechanical problems and be recertified frequently.

6.3.3 NIST Traceability and Certification of Flow-Rate Standards

The flow-rate standard used for flow-rate calibration should have its own certification and should be traceable to other standards for volume or flow rate which are themselves NIST traceable. A calibration relationship for the flow-rate standard, such as an equation, curve, or family of curves, should be established that is accurate to within 2 percent over the expected range of ambient temperatures and pressures at which the flow-rate standard is expected to be used. The flow-rate standard must be recalibrated or reverified and recertified at least annually.

The actual frequency with which this recertification process must be completed depends on the type of flow-rate standard—some are likely to be much more stable than others. The best way to determine recertification requirements is to maintain a control chart (a running plot of the difference or percent difference between the flow-rate standard and the NIST-traceable primary flow-rate or volume standard) for all comparisons. In addition to providing excellent documentation of the certification of the standard, a control chart also gives a good indication of the stability of the standard. If the two standard-deviation control limits are close together, the chart indicates that the standard is very stable and could be recertified less frequently. (The minimum recertification

frequency is 1 year.) On the other hand, if the limits are wide, the chart would indicate a less stable standard that should probably be recertified more often. A particularly unstable standard may be unusable no matter how often it is recertified.

6.3.4 Outline of Generic Flow-Rate Calibration Procedure

A fully detailed, EPA-approved flow-rate calibration procedure, tailored specifically for each commercially available PM_{2.5} sampler, is contained in the operations manual associated with each sampler that is designated as a reference or equivalent method under 40 CFR Part 53. That sampler-specific procedure should be followed carefully and thoroughly, in conjunction with the guidance in this section, to calibrate the sampler. The following steps outline a general calibration procedure.

1. Before performing a flow-rate calibration, make sure that the sampler temperature and pressure sensors have been calibrated.
2. Equilibrate the selected flow-rate calibration device with ambient conditions according to guidance provided in Section 6.3.2.
3. Install a filter cassette with an unused 47-mm filter in the sampler. This filter should meet all specifications for PM_{2.5} sampling, but it does not need to be preweighed or postweighed. Discard this filter once calibration is complete.
4. Remove the inlet from the sampler. Place the flow calibration device on the sampler down tube using a flow adapter device if necessary. Ensure that any valves are open so that flow through the sampler is unrestricted.
5. Perform a leak check according to the manufacturer's operating manual prior to continuing with flow calibration. Perform any corrective actions necessary to successfully meet the leak-check acceptance criteria. Leak checks are discussed in more detail in Section 10.2.2.
6. Place the sampler in calibration mode according to the instructions in the manufacturer's operating manual.
7. Follow the instructions in the manufacturer's operating manual for performing the flow calibration.
8. Once calibration is complete, turn off the sampler pump, remove the filter from the filter cassette, remove the flow calibration device (and flow adapter device, if applicable), and replace the sampler inlet.
9. The sampler flow rate is now calibrated. Refer to Section 6.3.1 for flow-rate verification procedures.

6.4 Calibration of Sampler Temperature Sensors

A fully detailed, EPA-approved calibration procedure for temperature sensors, tailored specifically for each commercially available PM_{2.5} sampler, is contained in the operation's manual associated with each sampler that is designated as a reference or Class I equivalent method under 40 CFR Part 53. The specific procedure(s) should be followed carefully and thoroughly to calibrate the sampler's temperature sensors.

This section provides additional precautions, guidance, and information on temperature standards and calibration that may not be contained in the specific instruction manual procedure. The section also presents a generic outline of the general procedure used for calibration of the temperature measurement systems of commercially available samplers. This information should be used to augment the specific instruction manual procedure.

6.4.1 General Requirements and Guidance

1. Multipoint calibration followed by single-point verification of each sampler's temperature sensors must be performed periodically (see Section 6.6 for guidance on determining frequency) to establish traceability of subsequent temperature measurements to an authoritative temperature standard. Calibration with a temperature standard that is traceable to a NIST-traceable primary standard (see Section 6.3.3 regarding NIST traceability) transfers the traceability to the sampler's temperature sensors. Because the temperature affects the flow rate, NIST traceability is also required for the sampler's temperature measurements.
2. PM_{2.5} sampler manufacturers use various types of temperature sensors. The specific calibration standard and procedure used for calibration or verification of the sampler's temperature sensor may vary depending on the type of sensors used.
3. Multipoint calibration of the sampler's temperature measurement system must consist of at least three separate temperature measurements approximately evenly spaced over the sampler's operational temperature range of -30 to +45 °C. Both the ambient air and filter temperatures are monitored in order to assess filter temperature deviation from ambient temperature and to set a flag should the filter temperature rise more than 5 °C above ambient and stay there for 30 consecutive minutes. The ambient temperature may also be used for computation of the volumetric flow rate.
4. Section 4.3.5 of the EPA QA Handbook, Volume IV, for meteorological measurements (EPA 1995) contains detailed guidance on calibration of temperature sensors/measurement systems, including a reference to the manufacturer's manual for adjustment of signal conditioning circuits, calibration equipment and methods for calibrating both T and ΔT (change in T), as well as an example of a calibration report.
5. Ideally, temperature calibration takes place in the field to assess sensor measurement system response as it occurs in normal operation. Usually the sensor is removed from the sampler while leaving it connected to the rest of the sampler's temperature measurement system and the space formerly occupied by the sensor should be plugged to prevent any contamination of the sampling system and to prevent entry of ambient air to that space.
6. Verification of a sampler's temperature sensor response should consist of one temperature measurement made at the sampler's current or average temperature reading for the week. This one-point verification may be substituted for a three-point calibration, provided that a full three-point calibration is carried out upon initial installation of the sampler and at least once per year thereafter, and the temperature measurement system has met the ±2 °C accuracy requirement in the previous three-point calibration. A full three-point calibration must be carried out whenever a one-point verification indicates that the sampler's

temperature measurement system differs by ± 4 °C or more from the temperature measured by the temperature standard. The one-point verification must be repeated following the three-point calibration.

6.4.2 Temperature Calibration Standards

The operations manual associated with the sampler will identify one or more types of temperature standards recommended for calibration and provide a detailed calibration procedure for each type that is specifically designed for the particular sampler.

The EPA Quality Assurance Handbook, Volume IV (EPA 1995), Section 4.3.5.1, gives information on calibration equipment and methods for assessing response characteristics of temperature sensors. The ambient air and filter temperature sensors of a reference or Class I equivalent PM_{2.5} sampler are required to have a resolution of 0.1 °C and an accuracy of ± 2 °C over the range of -30 to 45 °C. The handbook describes how to prepare three stable thermal mass assemblies whose temperatures can be known to about 0.1 °C. The thermal mass assemblies can be as simple as insulated vacuum bottles (i.e., thermos bottles) containing pure water or ice, or they may be solid cylinders of aluminum metal. A good ASTM- or NIST-traceable mercury-in-glass thermometer is also needed. It and the PM_{2.5} sampler sensor are both immersed in the thermal mass, and allowed to equilibrate; the temperature readings are compared.

6.4.3 NIST Traceability and Certification of Temperature Standards

The temperature standard used for temperature calibration should have its own certification and be traceable to a NIST primary standard. A calibration relationship to the temperature standard (an equation or a curve) should be established that is accurate to within 2 percent over the expected range of ambient temperatures at which the temperature standard is to be used. The temperature standard must be recalibrated or reverified and recertified at least annually.

The actual frequency of recertification depends on the type of temperature standard; some are much more stable than others. The best way to determine recertification requirements is to keep a control chart.

Additional reference sources concerning temperature measurements and calibration are: "Liquid-in-Glass Thermometry" (NIST 1976); "Thermometer Calibration: A Model for State Calibration Laboratories" (NIST 1986); "NIST Measurement Services: Liquid-in-Glass Thermometer Calibration Service" (NIST 1988); and "The Calibration of Thermocouples and Thermocouple Materials" (NIST 1989).

6.4.4 Outline of Generic Temperature Calibration Procedure

Both the ambient air and filter temperature sensors should be calibrated once per year. The ambient air sensor is located inside the shielded fixture on the outside of the PM_{2.5} sampler and is easy to unfasten and remove for comparison to a transfer standard for temperature. It is possible to conduct the three-point calibration at the field site, although it may prove easier to remove the sampler to the laboratory to avoid weather problems and for convenience in preparing the temperature standards. On the other hand, the filter temperature sensor of Reference or Class I equivalent PM_{2.5} samplers is located in the open space just below the filter cassette. It is threaded through the wall of the filter assembly section of the sampler and removal of plastic or metal

fittings is required to remove the sensor and its associated wiring. It is recommended that this sensor be calibrated in the laboratory. (The temperature sensor housing, the sampler inlet, and the interior of the downtube can also be cleaned in the laboratory.) Be careful when removing the filter temperature sensor—do not gall the fittings since this could start an internal leak after installation. It is suggested that a sampler leak check be performed after reinstallation of the filter temperature sensor.

Several steps to follow in calibrating ambient air temperature sensors are given below. Make frequent reference to the operator's instruction manual for sampler-specific procedures and instructions.

1. Remove the ambient temperature sensor from the aspirated radiation shield so that it can be placed in a constant temperature bath while it is still connected to the sampler's signal conditioner.
2. Prepare a convenient container (such as an insulated vacuum bottle) for the ambient temperature water bath and the ice slurry bath. See Step 3 below. If complete immersion of the sensor is necessary, wrap it in plastic film so liquid can reach the point where the connecting wire(s) and the sensor interface. Use partial immersion when possible, thus keeping the interface dry. If immersion is to be avoided altogether, it will be necessary to use thermal masses of metal rather than those based on water. Refer to Section 4.3.5 of Volume IV of the EPA QA Handbook (EPA 1995). To further insulate the vacuum bottle, it can be positioned inside a larger 2-gal insulated container that has been modified to allow wires or cables to enter the top. Refer to Figure 4.3.5.3 of Volume IV of the EPA handbook.

Keep the temperature changes relatively small and make comparative measurements in this order: AMBIENT→COLD→AMBIENT→HOT→AMBIENT. The range of temperatures need be only as broad as that expected to contain all the ambient temperatures that will be experienced during the upcoming time period, generally a year. The range to be expected is, of course, locale-specific. HOT may have to be 120 °F for Phoenix, Arizona, but around 80 °F for a location such as Barrow, Alaska.

3. For the ambient bath, use an insulated bottle that was filled with tap or deionized water several hours earlier and allowed to equilibrate to ambient temperature. For the ice slurry, the ice should be made with distilled water and then crushed into pea-sized pieces and mixed with distilled water until an easily penetrable slurry state is reached. As long as ice is present in the slurry and the open end of the bottle is guarded from ambient air temperature fluctuations, the ice slurry temperature will be $0.0 \pm 0.1^\circ\text{C}$.
4. Wrap the sensor(s) and a thermometer tightly together with a rubber band. The thermometer bulb and the temperature sensor active site should be close together. Immerse the sensor and the attached thermometer in the ambient temperature bath. Use a cork or some other device to cover the open end of the insulated bottle and thus keep ambient air from circulating over the top surface of the water (or ice slurry mass). Wait at least 5 minutes for the ambient thermal mass and the sensor/thermometer to equilibrate. Wait at least 15 minutes for equilibration with the ice slurry before taking comparative readings.
5. For each thermal mass, in the order indicated in Step 2 above, make a series of five measurements, taken about a minute apart. Accurately read the meniscus of the thermometer.

Use magnification if necessary to see the meniscus; avoid parallax errors. If the measurements made support the assumption of equilibrium, then average the five readings and record the result as the sensor temperature relative to the thermometer for ambient and for 0.0 °C relative to the ice slurry.

6.5 Calibration of Sampler Pressure Sensor

Each reference or Class I equivalent PM_{2.5} sampler has a built-in atmospheric pressure sensor whose output is processed to allow control of the actual sampling flow rate to the design value of 16.7 L/min. This section gives information concerning the care of barometers, their principles of operation, and how an aneroid barometer can be made traceable to a mercury column Fortin barometer and then be used to field-check the readings provided by the sampler's pressure sensor. The operation or instruction manual must be consulted for a sampler-specific information on how to make adjustments to calibrate the pressure sensor.

6.5.1 General Requirements

1. As required in 40 CFR Part 50, Appendix L, the sampler shall have the capability to measure the barometric pressure of the air surrounding the sampler over a range of 600 to 800 mm Hg. This measurement shall have a resolution of 5 mm Hg and a NIST-traceable accuracy of ± 10 mm Hg.
2. According to ASTM standard D 3631 (ASTM 1977), a barometer can be calibrated by comparing it with a secondary standard traceable to a NIST primary standard.
3. The Fortin mercurial type of barometer works on fundamental principles of length and mass and is therefore more accurate but is more difficult to read and correct than other types. By comparison, the precision aneroid barometer is an evacuated capsule with a flexible bellows coupled through mechanical, electrical, or optical linkage to an indicator. It is potentially less accurate than the Fortin type but can be transported with less risk to the reliability of its measurements and presents no damage from mercury spills. The Fortin type of barometer is best employed as a higher quality laboratory standard which is used to adjust and certify an aneroid barometer in the laboratory. The certified aneroid barometer can then be taken in the field and used to verify the readouts from the transducer type pressure sensor in the sampler. The sampler sensor can be left in the sampler during the comparison, since atmospheric pressures are equivalent within a 100-m horizontal distance and a 0.5-m vertical distance. If there is a discrepancy, the aneroid barometer should be recompared to the Fortin type upon return to the laboratory. A discrepancy with the sampler sensor could indicate an offset in the sampler's pressure sensor adjustment.
4. Protect all barometers from violent mechanical shock and explosively sudden changes in pressure. A barometer subjected to either of these events must be recalibrated. Maintain the vertical and horizontal temperature gradients across the instruments at less than 0.1 °C/m. Locate the instrument so as to avoid direct sunlight, drafts, and vibration.

6.5.2 Calibration Procedures

6.5.2.1 Fortin Type Barometer Readings

1. Read the temperature from the thermometer attached to the barrel to the nearest 0.1 °C.
2. Lower the mercury level in the cistern until it clears the index pointer. Raise the level slowly until a barely discernible dimple appears on the surface of the mercury.
3. Tap the barrel near the top of the mercury column.
4. Set the vernier so that the base just cuts off light at the highest point of the meniscus and carefully avoid parallax error.
5. Read the height of the mercury column from the barometer in the manner appropriate to the vernier scale used to the equivalent of the nearest 0.1 mm Hg. Apply appropriate corrections for temperature and gravity as described in the barometer instruction booklet.

6.5.2.2 Aneroid Type Barometer

1. Always use and read an aneroid barometer when it is in the same position (vertical or horizontal) as it was when calibrated.
2. Immediately before reading the scale of an aneroid barometer with mechanical linkages, tap its case lightly to overcome bearing drag.
3. Read the aneroid barometer to the nearest 1 mm Hg.

6.6 Calibration Frequency

1. The sampler's flow rate measurement system must be recalibrated after electromechanical maintenance or transport of the sampler. Be sure to check the temperature and pressure measurement systems also.
2. The sampler's flow rate measurement system should be recalibrated whenever there is any indication that the system is inaccurate or operating abnormally.
3. For routinely operated samplers that are performing properly, the sampler's flow rate measurement system should be recalibrated at periodic intervals not to exceed 1 year. A good way to determine an appropriate recalibration frequency for each sampler is to keep a control chart (a running plot of the difference (or percent difference) between the sampler's flow rate measurement and the flow rate measurement of the NIST-traceable flow rate standard) for all calibrations, audits, and flow checks. Such a chart also alerts the operator should the performance of the flow rate measurement system degrade to such an extent that repairs are required.
4. Calibration frequency for the temperature and pressure sensors should also be set based on such control charts or equivalent operational experience.

7.0 Filter Preparation and Analysis

7.1 Overview

The accuracy of a PM_{2.5} sampling program depends on several factors. A primary consideration is the analytical laboratory staff's attention to detail and balance technique. This section offers guidelines to enhance the accuracy of laboratory operation and, hence, the PM_{2.5} mass concentration and any additional chemical qualitative and quantitative determinations.

Microbalance resolution and repeatability requirements and filter media requirements are given in Section 4.3. Laboratory activities are summarized in Table 7-1.

7.2 Microbalance Environment

Gravimetric analysis of the filters is performed with a microbalance with a readability of 0.001 mg (1 µg) and a repeatability of 1 µg. Because of the greater sensitivity needed for measuring microgram-range weights or weight differences, microbalances are vulnerable to relatively small changes in physical environmental conditions, such as vibration, electrostatic charge buildup, temperature, or relative humidity. Features to offset the effect of these variables on the measurements may be integrated into the design of the microbalances, or they may be offered as options.

Each microbalance used in the weighing procedures must be identified by a balance number. Make sure that the microbalance has been calibrated (at least annually) and maintained according to the manufacturer's recommendations. If it is out of calibration, have the microbalance calibrated according to the manufacturer's directions.

If possible, locate the microbalance in a climate-controlled, draft-free room or chamber (preferably dedicated to the use of balances and employing a high-efficiency particulate air [HEPA]-filtered air supply system) to minimize human traffic effects and to stabilize the conditions of the weighing environment. Dust contamination can be minimized by clean room measures such as cleaning the weighing area daily, installing sticky floor covering on the entrance(s) to the weighing area, and wearing clean lab clothing over anything exposed to uncontrolled environments. Ideally, a small chamber can be used because it is easier and cheaper to control the environment in a smaller space.

If it is not possible to locate the microbalance in a climate-controlled, draft-free room or chamber, the following general guidelines should be followed to control environmental factors that may affect microbalance performance:

- Locate the microbalance away from potential sources of drafts such as doors, windows, aisles with frequent traffic, ventilation ducts, and equipment with fans or moving parts.
- Locate the microbalance out of direct sunlight and away from local heating or cooling sources such as open flames, hot plates, water baths, ventilation ducts, windows, and heat-producing lamps.
- Locate the microbalance on a sturdy base (ideally, a stone weighing table) and away from any equipment that produces vibrations. If these arrangements are not possible, isolate the

TABLE 7-1. FILTER PREPARATION AND ANALYSIS CHECKS

Activity	Method and frequency	Requirements	Action if the requirements are not met
Microbalance		Resolution of 1 µg, repeatability of 1 µg	Obtain proper microbalance
Microbalance environment		Climate-controlled, draft-free room or chamber or equivalent	Modify the environment
Mass reference standards	Working standards checked every 3 to 6 months against laboratory primary standards	Standards bracket weight of filter, individual standard's tolerance less than 25 µg, handle with smooth, nonmetallic forceps	Obtain proper standards or forceps
Filter handling	Observe handling procedure	Use powder-free gloves and smooth forceps. Replace ²¹⁰ Po antistatic strips every 6 months	Discard mishandled filter or old antistatic strip
Filter integrity check	Visually inspect each filter	No pinholes, separation, chaff, loose material, discoloration, or filter non-uniformity	Discard defective filter
Filter identification	Write filter number on filter handling container, sampler number on protective container, and both numbers on laboratory data form in permanent ink	Make sure the numbers are written legibly	Replace label or correct form
Presampling filter equilibration	Determine the correct equilibration conditions and period (at least 24 hours) for each new lot of filters. Observe and record the equilibration chamber relative humidity and temperature; enter to lab data form.	Check for stability of laboratory blank filter weights. Weight changes must be <15 µg before and after equilibration. Mean relative humidity between 30 and 40 percent, with a variability of not more than ±5 percent over 24 hours. Mean temperature should be held between 20 and 23 °C, with a variability of not more than ±2 °C over 24 hours.	Revise equilibration conditions and period. Repeat equilibration

(continued)

TABLE 7-1 (continued)

Activity	Method and frequency	Requirements	Action if the requirements are not met
Initial filter weighing	Observe all weighing procedures. Perform all QC checks	Neutralize electrostatic charge on filters. Wait at least 60 seconds after balance indicates a stable reading to record value.	Repeat weighing
Internal QC	After approximately every tenth filter, rezero the microbalance and reweigh the two working standards. Weigh three laboratory filter blanks. Periodically reweigh several filters (duplicate weighing).	The working standard measurements must agree to within 3 µg of the certified values (i.e., three times the microbalance's reproducibility). The blank measurements must agree to within 15 µg (i.e., three times the reproducibility for weighing unexposed filters).	Notify QC supervisor
Postsampling inspection, documentation, and verification	Examine the filter and field data sheet for correct and complete entries. If sample was shipped in a cooled container, verify that low temperature was maintained.	No damage to filter. Field data sheet complete. Sampler worked OK.	Discard filter. Void sample. Notify QC supervisor.
Postsampling filter equilibration	Equilibrate filters for at least 24 hours. Observe and record the equilibration chamber relative humidity and temperature; enter to lab data sheet.	Mean relative humidity between 30 and 40 percent, with a variability of not more than ±5 percent over 24 hours. Mean temperature should be held between 20 and 23 °C, with a variability of not more than ±2 °C over 24 hours.	Repeat equilibration
Postsampling filter weighing	Observe all weighing procedures. Perform all QC checks.	Neutralize electrostatic charge on filters. Wait 30 to 60 seconds after balance indicates a stable reading before recording data.	Repeat weighing

microbalance from such equipment by placing a stabilizing slab under the microbalance and/or by placing composite vibration damping pads at three points under the microbalance's

legs or under the stabilizing slab. Placing the pads at only three points eliminates the rocking associated with pads that are placed at four points.

- Ensure that the microbalance's base is sufficiently level to permit leveling of the microbalance according to the manufacturer's instructions.

7.3 Mass Reference Standards

Mass reference standards should be in the range of 0 to 200 mg, given that the mass range of typical 47-mm filter weights is from 110 to 160 mg. They must be certified as being traceable to NIST mass standards (see ASTM 1993b; Harris 1993; Kupper 1990). Additionally, they must have an individual tolerance of no more than 0.025 mg. Examples of mass reference standards that meet these specifications are ANSI/ASTM Classes 1, 1.1, and 2. The mass reference standards must be recalibrated on a regular basis (e.g., yearly) at a NIST-accredited State weights and measures laboratory or at a calibration laboratory that is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST (Harris 1994; White 1997). The recalibration frequency should be determined from records of previous recalibrations of these standards.

Note that the microbalance's resolution and repeatability are better than the tolerance of the most accurate classes of mass reference standards. Accordingly, the accuracy of the gravimetric analysis is limited by the tolerance of the standards rather than by the microbalance's characteristics.

Two separate sets of mass reference standards are recommended. Working calibration standards should be used for routine filter weighing and should be kept next to the microbalance in a protective container. Laboratory primary standards should be handled very carefully and should be kept in a locked compartment. The working standards should be compared to the laboratory primary standards every 3 to 6 months to check for mass shifts associated with handling or contamination. The current masses of the working standards as compared to the laboratory primary standards should be recorded in a laboratory notebook and used to check the calibration of the microbalance.

Always use smooth, nonmetallic forceps for handling mass reference standards. The standards are handled only with these forceps, which are not used for any other purpose. Mark these forceps to distinguish them from the forceps used to handle filters. Handle the standards carefully to avoid damage that may alter their masses.

7.4 Filter Handling

Careful handling of the filter during sampling, equilibration, and weighing is necessary to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters. Whenever filters are handled, the analyst must wear gloves that are powder-free and that act as an effective contamination barrier. Gloves that are packed in a box can carry an electrostatic charge. It is a good practice to discharge them by touching a good electrical ground after putting them on. The filters must be handled carefully with smooth, nonserrated forceps that are used only for that purpose. Mark these forceps to distinguish them from the forceps used to handle mass reference standards. These precautions reduce the potential effect from body moisture or oils contacting the

filters and subsequently affecting the measured weights.

In the laboratory, each filter should be transferred from its sealed manufacturer's packaging to a filter-handling container, such as a glass or plastic petri dish, to reduce the risk of contamination. The filter should remain in this container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter must have a unique identification number. A label that lists the filter number must be attached to the filter-handling container. It is recommended that each microbalance be assigned a block of filter numbers to be processed and used sequentially. Assign a filter identification number and take extreme care to avoid mistakenly assigning the same number twice or omitting a number.

If sufficient resources are available, bar coding for filters can be introduced. Bar code readers and printers for microbalances are already generally available and can significantly improve the efficiency of filter inventory tracking and processing.

7.5 Filter Integrity Check

All filters must be visually inspected for defects before the initial weighing. A filter must be discarded if any defects are found. Any lot of filters containing a high number of defects should be returned to the supplier. Specific defects to look for are the following:

1. **Pinhole**—A small hole appearing (a) as a distinct and obvious bright point of light when examined over a light table or screen or (b) as a dark spot when viewed over a dark surface.
2. **Separation of ring**—Any separation or lack of seal between the filter and the filter border reinforcing the ring.
3. **Chaff or flashing**—Any extra material on the reinforcing, polyolefin ring or on the heat seal area that would prevent an airtight seal during sampling.
4. **Loose material**—Any extra loose material or dirt particles on the filter.
5. **Discoloration**—Any obvious discoloration that might be evidence of contamination.
6. **Filter nonuniformity**—Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity or density across the face of the filter.
7. **Other**—A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

7.6 Filter Conditioning

Filters should be conditioned or equilibrated immediately before both the pre- and postsampling weighings. Filters must be conditioned for at least 24 hours to allow their weights to stabilize before being weighed.

Mean relative humidity should be held between 30 and 40 percent, with a variability of not more than ± 5 percent over 24 hours. Mean temperature should be held between 20 and 23 °C, with a variability of not more than ± 2 °C over 24 hours. Relative humidity and temperature must be measured and recorded on a daily basis during equilibration (either manually or by

hygrothermograph).

An air-conditioned room may be used as a conditioning chamber, if relative humidity and temperature can be maintained within the above specifications while filters are conditioning. If the room's heating and ventilation system cannot maintain relative humidity within these specifications, chemical or electronic methods must be used to control relative humidity. See ASTM (1993a) for information about maintaining constant relative humidity by means of saturated saltwater (e.g., magnesium chloride) solutions in an airtight chamber. Chambers with electronic humidity and temperature controllers are available from laboratory supply firms. Conditioning chamber malfunctions, discrepancies, and maintenance activities should be recorded in a logbook or a laboratory notebook.

Within the conditioning chamber, the filters should be placed on a covered rack or open-sided cabinet that will allow air circulation over the filters while reducing the chance that airborne material inside the chamber will settle onto the filters.

Filters should be conditioned in their filter-handling containers. Label, if possible, both the container's lid and bottom half. The lid must be removed during conditioning. Place the lid beneath the bottom half of the container to be certain that no filter mix-up occurs. To improve filter inventory control, care should be taken to stack the filters in the chamber in numerical order so that the analyst can more easily weigh the filters in numerical order.

Researchers in the desert western and southeastern portions of the United States have found that some Teflon[®] filters exhibit a loss of weight for a period of time after they are removed from their original shipping containers. The magnitude of weight loss varies from batch to batch and may be due to loss of volatile components from the polyolefin support ring. In the desert West, weight loss of up to 150 μg has been observed (Desert Research Institute 1994). Some filters require at least 6 weeks to equilibrate.

In the Southeast, filter weight stability experiments were done as part of EPA's research to develop the volatility test now included in 40 CFR Part 53.66 of the revised requirements for designation of reference and equivalent methods for $\text{PM}_{2.5}$ (Eisner 1997). Small but still relatively significant (i.e., from 0 to 45 μg) weight losses were observed. These experiments showed that the problem could be addressed by active conditioning (e.g., forced, HEPA-filtered air for 1-hour duration) instead of passive conditioning. The active conditioning was conducted with each filter sitting in the bottom of an open petri dish. Consecutive 4-hour periods of active conditioning of filters did not change the weight by more than $\pm 5 \mu\text{g}$.

Note: Typically, filters come packed together in large groups or in a container with separators. This package is usually contained inside another clear, reclosable plastic package, which may, in turn, be inside a box used in shipping. The less time that each filter is exposed to the conditioning environment, the less likely that its weight will be stable by the end of a conditioning period.

New filters should be removed individually from their sealed packages, placed in their own filter-handling containers (uncovered petri dish) and conditioned for a sufficient time to allow their weight to stabilize before use. Analysts may need to determine the conditions and time period needed to stabilize the weights for each new lot of filters. To determine this, take the two bottom and two middle filters from each pack of new filters as it is opened and expose each in a separate container in the conditioning chamber. Weigh the filters before and after the conditioning period.

If the weight change exceeds 15 μg (i.e., three times the reproducibility for weighing unexposed filters), the equilibration conditions and equilibration period must be modified. Conduct additional tests with unconditioned filters under the modified conditions and time periods until the weight change is less than 15 μg between weighings. Condition all other filters (separately) in the lot accordingly.

The stability of filter weights can be determined by assigning three filters from each new lot to be laboratory blanks. After an initial conditioning, these filters are stored in the conditioning chamber and reweighed in all subsequent weighing sessions involving filters from that lot. These measurements are recorded in the QC notebook (see Figure 7.1) or database. Analysis of long-term trends in the data via control chart plots can reveal whether the weight has stabilized. If weight changes are observed, the conditioning environment and conditioning duration can be modified to minimize such changes.

7.7 Electrostatic Charge Neutralization

Electrostatic charge buildup will prevent a microbalance from operating properly. Static charge is the accumulation of electrical charges on the surface of a nonconductive material. Common symptoms of this problem include noisy readout, drift, and sudden readout shifts. To reduce static charge within the balance, it may be necessary to place a radioactive antistatic strip containing a very small amount (i.e., 500 picocuries) of ^{210}Po in the weighing chamber. It may also be necessary to place each filter on an antistatic strip before it is weighed. See Engelbrecht et al. (1980), Hawley and Williams (1974), and Weil (1991) for more information about electrostatic charge and how to minimize its effects.

^{210}Po antistatic strips are used to reduce electrostatic buildup in the microbalance's weighing chamber and on individual filters by charge neutralization. They will neutralize electrostatic charges on items brought within an inch of them. These antistatic strips are safe, commonly available, and inexpensive. ^{210}Po has a half-life of 138 days. Change the antistatic strips annually and dispose of the old strips according to the manufacturer's recommendations. Antistatic solutions are available for coating (and, at appropriate and relatively infrequent intervals, recoating) the interior and exterior nonmetallic surfaces of the weighing chamber. This coating facilitates the draining of electrostatic charges away from these surfaces (by making them conductive) and to a common ground to which the metallic conductive surfaces are connected. Earth-grounded conductive mats may also be placed on the weighing table surface and beneath the analyst's shoe surfaces to reduce electrostatic charge buildup.

Do not assume that grounding eliminates all electrostatic buildup because the electrical ground may not be perfect. Even though a filter weight might stabilize within 30 to 60 seconds and no weight drift is observed during that period, the microbalance may still be influenced by some

Filter Lot Number C20102		Analyst J. Armstrong	
Balance Number A44603		QC Supervisor R. Vanderpool	
Analysis Date	Zero (Tare) Check Weight (mg)	Working Standard 1 Weight (mg)	Working Standard 2 Weight (mg)
6/30/97	0.000	199.999	1.996
8/13/97	0.000	200.001	1.997
Analysis Date	Laboratory Blank 1 Weight (mg)	Laboratory Blank 2 Weight (mg)	Laboratory Blank 3 Weight (mg)
6/30/97	136.546	129.999	130.633
8/13/97	136.550	130.006	130.645

Figure 7.1. Example laboratory internal QC log.

electrostatic buildup. It may still be necessary to repeat the neutralization procedure and to use antistatic strips inside the weighing chamber.

Charge neutralization times may need to be longer than 60 seconds for sampling situations in which (1) a high amount of charge has developed on collected particles due to their origin or (2) the particle loading on a filter is large. Examples of atmospheres that might be expected to contain a higher quantity of charged particles include air containing particles generated by mechanical means and air through which lightning has passed.

Electrostatic charge buildup becomes greater as the air becomes drier. A 60-second charge neutralization may be sufficient in ambient indoor air conditioned to 37 percent relative humidity and 23 °C but not in 17 percent relative humidity and 23 °C in the desert. This latter environment may require that the filter sit for more time on the antistatic strip. The longer neutralization period may have to be performed inside the weighing chamber or in a second small chamber used only for charge neutralization.

7.8 Presampling Filter Weighing (Tare Weight)

The reference method (EPA 1997) requires that the presample filter weighing be conducted within 30 days of the sampling period. The microbalance must be located in the same controlled environment in which the filters are conditioned. The filters must be weighed immediately following the conditioning period without intermediate or transient exposure to other conditions or environments.

This section presents procedures specific to a common commercially available microbalance. Calibration, QC checks (and acceptance tolerances), and operational procedures may have to be adapted for use with other microbalance models.

Researchers have found that the reproducibility of mass measurements for unexposed filters based on replicate weighings is typically 3 to 5 µg/filter (Desert Research Institute 1994). The precision on exposed filters is typically 5 to 8 µg/filter. The repeatability for filters with loadings heavier than 1 mg/cm² of filter surface area may approach ±2 percent of the loading. The repeatability for net mass filter loadings is the square root of the sum of the squares of the pre- and postsampling repeatability (typically 6 to 9 µg/filter).

These steps should be followed during the presampling filter weighing:

1. Clean the microbalance's weighing chamber with a fine brush, if necessary. Avoid using pressurized gas, which may blow damaging debris and oils into the microbalance's mechanism. Recoat the interior and external nonmetallic surfaces of the chamber with an antistatic solution, if necessary. Clean the surfaces near the microbalance with antistatic solution- or methyl alcohol-moistened disposable laboratory wipes. Clean the standard forceps with a lint-free cloth and the filter forceps with the moistened wipes. Make sure the forceps are thoroughly dry before use. Even a small amount of moisture can cause a significant measurement bias.
2. To ensure maximum stability, it is recommended that the microbalance be turned on at all times. This procedure enables the microbalance to be operational at any time and eliminates

the need for a warmup period before analyses are performed. Newer microbalances are always turned on (except for their displays) when they are plugged in.

3. Zero (i.e., tare) and calibrate the microbalance according to the manufacturer's directions. Record the tare weight on the laboratory data form (see Figure 7.2) and in the laboratory notebook or database (see Figure 7.1). Many newer microbalances calibrate themselves automatically or require only a key to be pressed to calibrate themselves.
4. Using smooth, nonserrated, nonmetallic forceps, weigh two working mass reference standards (for example, a 100 or 200 mg and a 1 or 2 mg standard) as a QC check. Handle the working standards carefully to avoid damage that may alter their masses. Recheck the standards annually or after any incident of rough handling against the laboratory's primary standard weights (ASTM Class 1 or 1.1 or OIML Class E1 or E2 standards). The 100 or 200 mg standard approximates the mass of a blank or a loaded filter. Wait until the microbalance's display has remained steady for 30 to 60 seconds or until the microbalance indicates that a stable reading has been obtained. Record the certified and measured values of these standards on the laboratory data form and in the laboratory notebook or database.

If the certified and measured values of a working standard disagree by more than $3 \mu\text{g}$ (i.e., three times the microbalance's repeatability), reweigh the working standard. If the two values still disagree, contact the laboratory's QC supervisor, who may direct the analyst to (1) troubleshoot or repair the microbalance or (2) recertify the working standard against the laboratory primary standard.

5. Record the relative humidity and temperature of the conditioning chamber on the laboratory data form and in the laboratory QC notebook or database.
6. Laboratory blank filters and the current sampling interval's field blank filters should be weighed at least once in each weighing session. If many filters are weighed, you may want to weigh the set of laboratory blanks more than once. Laboratory blank filters are kept in the laboratory in a protective chamber or container and should be reweighed during each weighing session as a QC check. A new set of three laboratory blanks should be established for each distinct filter lot. Field blank filters are transported to the sampling site, momentarily installed in the sampler, removed, and stored in their protective containers, inside the sampler's case at the sampling site until the exposed filters are retrieved for postsampling weighing. In the case of sequential samplers, field blanks should be placed in unused sampler slots, kept there during the sampling interval, and retrieved with the exposed filters. This presumes the slots are available and the sampler can be programmed to not sample the field blank filter. If this cannot be done, follow the procedure for the manual sampler. These field blanks will be reweighed during the final (postsampling) filter weighing as a QC check and compared to their initial weights.

At the time of the presampling filter weighing, weigh three laboratory filter blanks and three field filter blanks and record the values in the laboratory data form and the laboratory QC notebook or database. Return the laboratory blank filters to their protective filter holders and

Filter Lot Number C20102		Analyst J. Armstrong		
Balance Number A44603		QC Supervisor R. Vanderpool		
Presampling Filter Weighing		Date 6/30/97	RH 43	Temp 76
Postsampling Filter Weighing		Date 8/13/97	RH 48	Temp 75
Sampler ID	Filter Number ^a	Presampling Mass (mg)	Postsampling Mass (mg)	Net Mass Filter Loading (mg)
	0	0.000	0.000	—
	200 mg	199.999	200.001	0.002
	2 mg	1.996	1.997	0.001
A1	R-691	139.293	139.727	0.434
A2	R-692	136.020	136.455	0.435
A3	R-693	135.818	136.260	0.442
B1	R-694	131.456	131.905	0.449
B2	R-695	137.508	137.973	0.465
B3	R-696	136.098	135.554	0.456
C1	R-697	131.029	131.483	0.454
C2	R-698	125.175	125.641	0.466
C3	R-699	131.165	131.633	0.468
Lab Blank 1	D-110	136.546	136.550	0.004
Lab Blank 2	D-111	129.999	130.006	0.007
Lab Blank 3	D-112	130.633	130.645	0.012
Field Blank 1	R-700	130.896	130.904	0.008
Field Blank 2	R-701	128.339	128.345	0.006
Field Blank 3	R-702	130.929	130.936	0.007

Figure 7.2. Example laboratory data form.

store them in the conditioning chamber. Handle the field blank filters in the same manner as filters that will be used for sampling.

7. Weigh the filters. Operate the balance according to the balance manufacturer's directions. Take the filter from its filter-handling container (petri dish or equivalent) by gently slipping the filter-handling forceps under the outer polyolefin support ring. Hold the filter only by the ring. Place the filter, reinforcing ring side up, on a ^{210}Po antistatic strip for 30 to 60 seconds. The antistatic strip should be inside the weighing chamber or as close to the chamber door as is practical. Immediately transfer the filter to the microbalance's pan and close the weighing chamber door. After the microbalance's display has remained steady for at least 60 seconds or until the microbalance indicates that a stable reading has been obtained, record the balance number, the sampler number the filter is intended to be used with, the filter number, the filter lot number, and the filter's tare weight (presampling mass) on the laboratory data form.
8. After approximately every tenth filter weighing, the analyst should rezero the microbalance and reweigh the two working standards. Record the zero and working standard measurements on the laboratory data form and the laboratory QC notebook or database. If the zero and working standard measurements disagree from the first measurements of the day by more than $3\ \mu\text{g}$ (i.e., three times the microbalance's reproducibility), repeat the zeroing process and reweigh the working standards. If the two measurements still disagree, contact the laboratory's QC supervisor, who may direct the analyst to (1) reweigh the previously weighed filters and/or (2) troubleshoot or repair the microbalance and repeat the weighing session.

Newer microbalances are so easily rezeroed that analysts may elect to rezero before each weighing.

9. Any unused filter whose weight is outside the normal range (i.e., 110 to 160 mg) must be investigated. If there is a consistent negative replication ($>15\ \mu\text{g}$) for laboratory blank filters, it is usually a sign that the filters have not equilibrated long enough. In this case, notify the QC supervisor.
10. Return the filter to the filter-handling container, replace the lid, and return it to storage.
11. When the time comes for the filters to be taken to the sites (must be within 30 days of the initial weighing), install each filter in a filter cassette, and put the filter/cassette assembly into a protective container for transport to the sampler (see Figure 7.3). Attach a label with the sampler number and the unique filter number to the outside of the protective container. This label will also be used to identify the upcoming sample run date. Record the sampler number, sample date, and filter number on the $\text{PM}_{2.5}$ Sampler Run Data Sheet (see Figure 8.1). Double-check the entries in the laboratory data form (Figure 7.1). The sample will have to be invalidated if it cannot be reconciled with the correct sampler and filter identification numbers. Prepare several extra filters in case a filter is invalidated during the installation process.
12. If filters are to be mailed, the field operator should be supplied with reinforcing envelopes or some other means (in addition to the protective container) to protect exposed filters during their shipment back to the analytical laboratory.

7.9 Postsampling Documentation and Inspection

Upon receipt of the sample from the field, the analyst should follow these steps:

1. Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration (e.g., average flow rate, ambient temperature and barometric pressure, and elapsed time) are provided. If data are missing or unobtainable from a field operator or if a sampler malfunction is evident, discard the filter and record in the laboratory data form that the sample has been voided and the reason. Notify the QC supervisor.
2. If the shipment was to be kept cold, verify that the temperature of the cooler's interior was maintained at the desired point, usually less than 4 °C. This can be done by quickly reading the min/max thermometer that was placed in the container at the time of shipment, or inspecting the temperature-indicating liquid crystal strips, or by reading a thermometer or thermistor probe placed inside the shipping container adjacent to the sample(s) soon after its arrival at the laboratory. If the protective metal container is cold, allow it to warm to the filter conditioning environment's temperature before opening, to preclude water condensation on a cold filter. Remove the filter from its protective container and examine the container. If particulate matter or debris is found in the protective container after the filter has been removed, record notes on the laboratory data form that the sample has been voided and the reason. Save the filter for inspection by the QC supervisor.
3. Match the sampler number with the correct laboratory data form on which the original microbalance number, filter number, presampling filter weight, and other information were inscribed. Group filters according to the microbalance used to determine their initial tare weights. Initial separation of filters in this way will eliminate the risk of a measurement error that could result from the use of different microbalances for pre- and postsampling weighings.
4. Remove the filter from both the protective container and the filter cassette. Some cassettes may require special tools to disassemble them. Be very careful when removing the filter from the cassette. Be careful not to touch or otherwise disturb the filter and its contents. Transfer

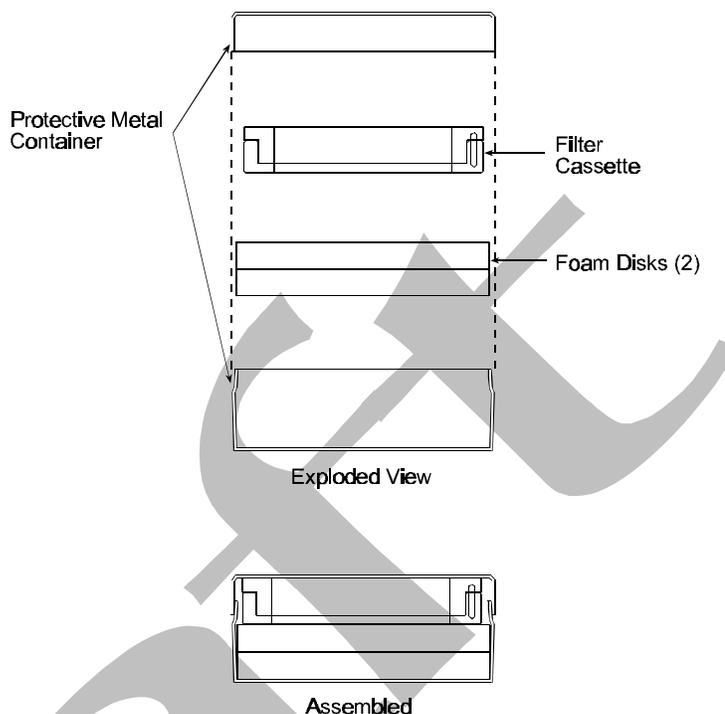


Figure 7.3. PM_{2.5} filter cassette and protective metal container.

the filter to a filter-handling container labeled with the corresponding filter number. Place the used filter in the container “dirty-side” up. Keep the particles from contact with the walls of the container. The filter must be handled with clean, smooth forceps and must not be touched by hands. Inspect the filter for any damage that may have occurred during sampling. If any damage is found, void the sample, and record on the laboratory data form that the sample has been voided and why. Retain the filter for inspection by the QC supervisor.

5. Transfer the filter in its filter-handling container to the conditioning chamber.
6. Allow the filter to condition for not less than 24 hours.

7.10 Postsampling Filter Weighing (Gross Weight)

Both the pre- and postsampling filter weighing must be carried out on the same analytical balance, and preferably by the same analyst. Use an effective technique to neutralize static charges on the filter. The postsampling conditioning and weighing should be completed within 240 hours (10 days) after the end of the sampling period, unless the filter is maintained at 4 °C or less during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 days.

The following steps should be followed during postsampling filter weighing.

1. Group filters according to the microbalance used for pre-weighing and by their filter numbers. Reweigh each filter on the same microbalance on which its presampling weight was obtained.
2. Repeat Steps 1 through 10 in Section 7.8
3. If the pre- and postsampling weights for the laboratory and field filter blanks disagree by more than 15 µg (i.e., three times the reproducibility for unexposed filters), repeat the measurements. If the two measurements still disagree, contact the laboratory’s QC supervisor, who may direct the analyst to (1) reweigh the previously weighed filters and/or (2) troubleshoot or repair the microbalance, then reweigh.
4. If the filter will receive further analysis, return it to the filter-handling container and note on the container and the laboratory data form that additional analyses are required. Transfer the filter to the laboratory responsible for performing the additional analyses.

7.11 Calculation of Net Mass Filter Loading

A filter’s postsampling mass minus its presampling mass is the net mass loading for that filter. Record this value on the laboratory data form. Refer to Section 11.0 for the calculations required to compute and report ambient PM_{2.5} concentrations in µg/m³.

7.12 Additional PM_{2.5} Analysis Requirements

Section 2.8.1.5 of 40 CFR Part, Part 58, Appendix D states:

- (a) Within 1 year after September 16, 1997, chemical speciation will be required at approximately 25 PM_{2.5} core sites collocated at Photochemical Assessment Monitoring Station (PAMS) sites (one type 2 site per PAMS area) and at approximately 25 other core

sites for a total of approximately 50 sites. The selection of these sites will be performed by the Administrator in consultation with the Regional Administrator and the States. Chemical speciation is encouraged at additional sites. At a minimum, chemical speciation to be conducted will include analysis for elements, selected anions and cations, and carbon. Samples for required speciation will be collected using appropriate methods and sampling schedule in accordance with procedures approved by the Administrator.

(b) Air pollution control agencies shall archive PM_{2.5} filters for all other SLAMS sites for a minimum of 1 year after collection. These filters shall be made available for supplemental analyses at the request of EPA or to provide information to State and local agencies on the composition for PM_{2.5}. The filters shall be archived in accordance with procedures approved by the Administrator. Storage conditions will likely involve temperature control (4 °C or less), humidity control, and protection from light, vibrations, and dust sources.

7.13 Internal Quality Control

Keep a QC notebook or database (with disk backups), which will contain QC data, including the microbalance calibration and maintenance information, routine internal QC checks of mass reference standards and laboratory and field filter blanks, and external QA audits. These data will duplicate data recorded on laboratory data forms but will consolidate them so that long-term trends can be identified. It is recommended that QC charts be maintained on each microbalance and included in this notebook. These charts may allow the discovery of excess drift that could signal an instrument malfunction.

At the beginning of each weighing day, after the analyst has completed zeroing and calibrating the microbalance and measuring the working standard, weigh the three laboratory filter blanks established for the current filter lot and three field filter blanks from the most recently completed field blank study. After approximately every tenth filter weighing, the analyst should reweigh the two working standards and rezero the microbalance. Record the zero, working standard, and blank measurements in the laboratory data form and the laboratory QC notebook or database. If the working standard measurements differ from the certified values or the presampling values by more than 3 µg (i.e., three times the microbalance's reproducibility), repeat the working standard measurements. If the blank measurements differ from the presampling values by more than 15 µg (i.e., three times the reproducibility for unexposed filters), repeat the blank measurements. If the two measurements still disagree, contact the laboratory's QC supervisor, who may direct the analyst to (1) reweigh some or all of the previously weighed filters, (2) recertify the working standard against the laboratory primary standard, and/or (3) troubleshoot or repair the microbalance.

If more than one microbalance is used, the pre- and postsampling measurements of the filter must be made on the same microbalance.

The QC supervisor must certify on the laboratory data forms the acceptability of filter weighings and QC checks and the completeness of the data. The QC supervisor must sign each completed form. When bound together, these forms serve as a laboratory notebook.

8.0 Field Operation

8.1 Overview

Sampling operations provided in this section are independent of any one type of commercially available PM_{2.5} sampler. Because operational procedures may vary among sampler models, the manufacturer's instrument manual should be consulted before the sampler is put into operation. Sampling procedure checks are summarized in Table 8-1.

TABLE 8-1. SAMPLING PROCEDURE CHECKS

Procedure	Frequency and method	Requirements	Action if requirements not met
Filter installation	Visually check filter. Install filter cassette, and securely close filter holder.	Filters must be properly identified, tare-weighed, and undamaged.	Void the filter, and install substitute filter.
Sample validation and documentation	Visually check each sample and the sample data sheet for completeness.	Record sampling date, filter and sampler ID, station location, flow rates, sample time, and unusual conditions on data sheet.	Complete or correct documentation; if unavailable, void the sample.
Postsample inspection	Visually check each sample for tears, missing pieces, or leakage.	There should be no evidence of filter damage or sampler malfunction.	Void the sample; correct the cause of malfunction.
Leak checks	Check for leaks at least weekly.	Sampler leak check must be within parameters specified by manufacturer.	Determine cause of leak and correct. Calibrate the sampler.
Flow checks	Check flow rate at least weekly.	Sampler flow rate must be within ± 2 percent of the specified flow rate.	Determine cause of flow problem and correct. Calibrate the sampler.
Field blank check	Periodic; install, then immediately remove filter from sampler; store in protective container inside sampler case.	Rotate from sampler to sampler; three field blanks each sampling period; special requirements for sequential samplers.	Reassess filter handling techniques and storage conditions.

8.2 Activities to Perform Each Sampling Day

8.2.1 Beginning a Run

Care must be taken to ensure that a filter is clean and undamaged before it is installed in a PM_{2.5} sampler. A filter cassette should be kept in its protective metal container; damaged filters must be discarded.

The procedure used to install a filter in a PM_{2.5} sampler is presented below.

1. Mark the following on the PM_{2.5} Sampler Run Data Sheet (Figure 8.1):
 - Site designation and location
 - Sampler model, ID number, and filter ID number
 - Sample start date and time
 - Initial temperature and barometric pressure indicated by the sampler
 - Unusual conditions that may affect the results (e.g., subjective evaluation of pollution on that day, construction activity, weather conditions)
 - Signature.
2. Ensure the sampler is not operating. If the sampler is set to automatically begin operation, ensure that enough time is available to complete these setup procedures.
3. Open the filter holder assembly according to the manufacturer's instructions. Install the filter cassette containing the preweighed filter. Visually inspect the O-rings inside the filter holder to ascertain that they are present and secure. Do not sample without these O-rings installed, as the system will no longer be leak-free. Reinstall the filter holder and ensure that the fittings around the impactor housing and the filter assembly are secure.
4. The sampler is now ready to sample. If the sampler is not already set to energize at the beginning of the next sampling period, set the controls (according to the manufacturer's instructions) to do so.
5. Visually inspect the monitoring site and its equipment to ensure that all sampling components are ready for the next run day. Note any changes in the site surroundings, especially dust-producing activities.
6. Take and record independent measurements of ambient temperature and pressure, and ensure that the ambient temperature and pressure readings taken by the sampler are within 2.0 °C and 10 mm Hg of the independent readings, respectively. Be certain that the independent temperature sensor (thermometer or thermistor probe) is located side-by-side with the sampler's ambient temperature sensor. This will require that the sensor be placed in the louvers of the sampler's temperature screen and kept out of direct sunlight.
7. About 15 min after the sampler begins operating, record the indicated flow rate. If operator cannot be present, record this information later from the downloaded runtime data set.

	Yes	No
Questionable	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Void	<input type="checkbox"/>	<input checked="" type="checkbox"/>

PM_{2.5} Sampler Run Data Sheet

Site Identification **NC-723** Site Name **Research Triangle Park**

Sampler Model **O-A-2013** Sampler ID **A1** Filter ID **R691**

Start Date **8/8/97** Stop Date **8/8/97** Elapsed Time **24** hr

Start Time **0:00** Stop Time **24:00**

Initial Conditions:

Sampler: Indicated P_a: **752** mm Hg Indicated T_a: **24** °C

Indicated Q: **16.7** L/min

Other: P_a **754** mm Hg Measured using: **Field barometer #4**

T_a **24** °C Measured using: **ASTM thermometer**

Final Conditions:

Sampler: Indicated P_a: **758** mm Hg Indicated T_a: **23** °C

Indicated Q: **16.4** L/min

Indicated Q_{avg}: **16.7** L/min Indicated Q_{cv}: **0.3**

Volume sampled: **24** m³

Other: P_a **757** mm Hg Measured using: **Field barometer #4**

T_a **24** °C Measured using: **ASTM thermometer**

Filter Information:

Filter ID: **R691** Initial Weight: **139.2** mg Final Weight: **139.72** mg

93 **7**

Calculated Concentration: **18.08** µg/m³

Notes: _____

Setup Operator: J. Jones End Operator: A. Smith

Figure 8.1. Example PM_{2.5} sampler run data sheet.

8.2.2 Ending a Run

1. If it is possible for the operator to be present, prior to the end of the sampling period (while the sampler is still running), visually inspect the sampler readouts to ensure that the sampler is operating properly. Also check the sampler for any other obvious problems, such as a full water collection jar. If problems are identified, describe them on the sample run data sheet and take corrective actions before proceeding.
2. Record the following information on the sampler run data sheet:
 - Stop time and total elapsed time of the sample run
 - Final flow rate, average flow rate, coefficient of variation of the flow rate, and total volume sampled
 - Final indicated ambient temperature and barometric pressure
 - Independent measurements of ambient temperature and barometric pressure
 - Existing conditions that may affect the results
 - Any flags triggered by the sampler (e.g., power outage, flow rate variation)
 - Explanations for voided or questionable samples
 - Signature.
3. Download the runtime data for the completed run using a computer.
4. Carefully open the filter holder assembly according to the manufacturer's instructions. If the filter cassette comes apart or sticks to the upper housing during this process, close and gently reopen the assembly. Do not allow the filter to be touched by any foreign object (fingers, rain, etc.), shaken, or dropped. Place the filter cassette inside the appropriately marked protective metal container for storage and transport.
5. Inspect the interior of the filter housing and the sampler itself. Note any abnormalities on the sampler run data sheet.
6. Inspect the interior of the impactor housing and the exterior of the impactor well. These areas should be clean and dry. If necessary, note the condition on the sampler run data sheet and clean the areas with a lint-free wipe.
7. Without opening the impactor well, inspect the interior of the well. The filter should be intact, with enough diffusion oil to saturate but not submerge it. It is normal for a small mound of material to be present in the center of the bottom of the well (underneath the impaction jet). Note any abnormalities on the run data sheet and clean or replace the impactor well as necessary. Replace the well assembly in the impactor assembly and reinstall the assembly in the sampler.
8. If another sampling run will be performed, install a filter cassette according to the instructions in Section 8.1.1, above, and the manufacturer's instructions.
9. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the run data sheet.

8.2.3 Sample Validation

The following criteria have been established to assist the operator in determining whether or not a sample is valid:

1. **Timing:**

- All samplers must operate for at least 23 but not more than 25 hours (1,380 to 1,500 min).

2. **Flow Rates:**

The average flow rates must be within 2 percent of 16.67 L/min at actual conditions. If this limit is exceeded, investigate the cause. Use the following criteria as a basis for sample invalidation:

- Decreases in flow rate during sampling (due to mechanical failure) of more than 2 percent from the initial set point require a field calibration check. If the sampler's calibration check indicates that the sampler flow was not within ± 2 percent of the designed flow, the sample should be invalidated.
- If the sampler flow rate decreased because of heavy particulate loading on the filter, the sample should not be invalidated because the heavy loading may indicate an episodic situation that deserves study.
- Changes in flow rate calibration of more than 2 percent, as determined by a weekly field calibration check, will invalidate all samples collected since the last acceptable flow rate check. Recalibrate the sampler.

3. **Filter Quality:**

- Any filter that is obviously damaged (i.e., torn, frayed, or has pin holes) should be invalidated.

4. **Temperature:**

- Based on the data downloaded from the sampler for the run, the filter temperature must be no greater than 5 °C above the ambient temperature for at least 30 consecutive minutes, based on 5-min averages. Recalibrate or repair the temperature sensors if necessary.

8.2.4 Sample Handling

8.2.4.1 Handling of a Valid Sample

1. Promptly deliver the filter cassette in its protective metal container, accompanied by the completed run data sheet, to the analytical laboratory. Package and handle the samples to avoid vibrations that may dislodge particles. Do not allow the filter surfaces to touch any of the interior surfaces of the protective metal container. The metal container should be transported in an insulated container capable of maintaining a temperature of less than 25 °C (77 °F). If desired, the sample may be cooled to 4 °C by placing leak-proof ice substitutes inside the insulated container. Do not allow the protective metal container to become wet.

Place a min/max thermometer or a liquid crystal temperature sensor strip inside the shipping container to verify that the sample(s) is remained cool during its transit to the laboratory. Alternatively, a temperature sensor probe can be inserted in the shipping container when it arrives at the laboratory and a check can be made then. Handle field blank filters in the same manner.

8.2.4.2 Handling of an Invalid Sample

1. Complete as much of the run data sheet as possible and explain any omissions.
2. Mark "VOID" on the run data sheet and record in the site logbook.
3. Do not discard the filter.
4. Promptly deliver the filter cassette in its metal container (as if it were a valid sample) and the run data sheet to the analytical laboratory, where a final decision on sample validity will be made.

8.2.4.3 Handling of a Questionable Sample

If you are uncertain whether or not a sample should be voided, take the following steps:

1. Complete as much of the run data sheet as possible and explain any factors that may affect the sample validity.
2. Indicate the sample as questionable in the upper right corner of the run data sheet.
3. Record the sample as "Questionable" in the site logbook.
4. Promptly deliver the filter cassette in its metal container, as if it were a valid sample, and the run data sheet to the analytical laboratory, where a final decision on sample validity will be made.

8.3 Activities to Perform Every 5 Days

8.3.1 Impactor Well Replacement

For $PM_{2.5}$ samplers, replacement of the impactor well is strongly recommended after every 5 days of operation. Also empty water from the water trap, clean the interior, inspect the seals, and reinstall the trap. Replacement of the well is necessary to prevent the sampler from operating with an overloaded $PM_{2.5}$ impactor. When operating in areas with significantly higher than average ambient particulate concentrations, more frequent replacement of the wells may be necessary. Refer to Section 9.2.2 for impactor maintenance guidance.

Wells should be prepared in a controlled laboratory space, not in the field. Transport a installation-ready well to the sampling site in a protective case in an upright position. During transport, protect the well from excessive vibration, precipitation, direct sunlight, and other harmful environments. Replace the well during normal end-of-run activities and carefully transport the used well back to the laboratory. You may want to number or otherwise identify each well assembly and keep records of its use.

8.3.2 Operator's QC Field Calibration Check Procedure

For PM_{2.5} samplers, a field calibration check of the flow rate is recommended after every 5 days of operation. The purpose of this check is to track the sampler calibration stability. Control charts presenting flow check data (indicated vs. observed) should be maintained. These charts provide a quick reference of instrument flow rate drift patterns and indicate when flow limits (± 2 percent variation from the indicated or design condition flow rate) have been exceeded. The field check is made by installing a measuring device (which is traceable to NIST and is calibrated within the range of the flow rate) on the inlet of the sampler. Calibration procedures for the measurement device are referenced in Section 6.3.4. Be sure to conduct a leak-check test before conducting the flow-rate check.

Calibration checks of the sampler flow rate require that the instrument be running. The following flow check procedures are independent of any device. A variety of transfer standards may be used with this same procedure; however, the necessary apparatus and subsequent calculations to determine the sampler's flow rate will vary.

8.3.2.1 Field Check Apparatus

The following equipment is required for field calibration checks of temperature, pressure, and flow rate:

- A thermometer capable of accurately measuring temperature to the nearest ± 1 °C and referenced to a NIST standard or ASTM thermometer within ± 0.1 °C at least annually
- A barometer capable of accurately measuring ambient barometric pressure to the nearest ± 1 mm Hg and referenced to a NIST or ASTM barometer within ± 5 mm Hg at least annually
- An orifice device (or other acceptable flow measurement device) and calibration relationship
- The sampler's calibration information (equation)
- A clean flow check filter, installed in a cassette
- The PM_{2.5} sampler flow check data sheet (Figure 8.2) or logbook.

8.3.2.2 Leak Check

Before conducting QC field calibration checks of temperature, pressure, and flow rate, carry out a sampler leak check as follows.

1. Insert the clean filter (designated the "leak-check filter") into the sampler filter holder as described in the operating procedure in Section 8.1. Leak check filters should never be used for subsequent sampling. The same filter may be used for the leak check as was used for the field calibration check.
2. Remove the inlet and place the flow rate adapter on the top of the downtube (see Figure 10.3). Close the adapter valve to prevent air flow.
3. Perform the leak check procedure according to the specific instructions in the manufacturer's operating manual.

PM_{2.5} Sampler Flow Check Data Sheet		
		Date of Flow Check _____
		Time of Flow Check _____
Site Identification _____	Site Name _____	
Sampler Model _____	Sampler ID _____	Filter ID _____
Sampler Indicated P _a _____ mm Hg	T _a _____ °C	Other P _a _____ mm Hg T _a _____ °C
Unusual Conditions: _____		
Flow Check Device _____	S/N _____	Calibration Date _____
Flow Check Device Calibration Relationship: m: _____ b: _____ r: _____		
Measurement No.	Indicated Flow, L/min (Q _{ind})	Measured Flow, L/min (Q _{ref})
1		
2		
3		
4		
5		
Average		
Percent Difference from Reference Measured Flow Rate (a) _____		
(a) Percent Difference = [(Average Q _{ind} - Average Q _{ref})/Average Q _{ref}] · 100		
Percent Difference from Design Flow Rate (b) _____		
(b) Percent Difference = [(Average Q _{ind} - 16.7)/16.7] · 100		
Operator's Signature _____		

Figure 8.2. Example PM_{2.5} sampler flow check data sheet.

CAUTION

Open the flow-rate adapter's valve slowly to prevent a sudden rush of air into the system which could cause oil to splash from the impactor well onto the filter and other components.

4. If the sampler leak check is within acceptable limits as detailed in the operating manual, the sampler is operating properly and you may proceed with the temperature, pressure, and flow-rate checks. If the limit is exceeded, investigate and correct any malfunction. If necessary, recalibrate the sampler before sampling is resumed.
5. Turn off the sampler, remove the flow audit device, and replace the inlet.
6. Remove the leak-check filter from the filter holder. Do not re-use this filter for sampling.

8.3.2.3 Procedure for Field Calibration Check

1. Insert the clean filter (designated "flow-check filter") into the sampler filter holder as described in the operating procedure in Section 8.1. Flow-check filters should never be used for subsequent sampling.
2. Turn on the sampler and allow it to warm up to operating temperature (approximately 5 min).
3. Read and record the following information on the PM_{2.5} sampler flow-check data sheet (Figure 8.2):
 - Ambient temperature (T_a), °C (as indicated by the sampler's display)
 - Ambient temperature (T_a), °C (as indicated by the field-check thermometer or thermistor)
 - Ambient barometric pressure (P_a), mm Hg (as indicated by the sampler's display)
 - Ambient barometric pressure (P_a), mm Hg (as indicated by the field-check barometer)
 - Sampler ID number and model
 - Flow-check device serial number and calibration relationships
 - Date, location, and operator's signature.
4. Remove the inlet from the sampler downtube and replace it with the flow-check device. Allow time for the flow-check device to reach ambient temperature. See Figure 8-3.
5. Record the flow rate measured by the flow-check device and the actual flow rate indicated by the sampler. Repeat this process for a total of at least five measurements. Take the average of the recorded measured flow rates and the average of the indicated flow rates.
6. Using the above information, calculate the percentage difference as:

$$\% \text{ difference} = [(\text{indicated}_{\text{avg}} - \text{measured}_{\text{avg}}) / \text{measured}_{\text{avg}}] \cdot 100\%$$

7. If the sampler flow rate is within 98 to 102 percent of the measured flow rate (at actual conditions), **and** if the sampler flow rate is within 95 to 105 percent of the design flow rate of 16.7 L/min, the sampler is operating properly. If either limit is exceeded, repeat the leak-check procedure, as stated in Section 8.3.2.2. After doing so, investigate and correct any malfunction. If necessary, troubleshoot the sampler further and recalibrate it before sampling is resumed.
8. Turn off the sampler, remove the flow-check device, and replace the inlet.
9. Remove the flow-check filter from the filter holder.
10. Set up the sampler for the next sampling period according to the operating procedure in Section 8.1.

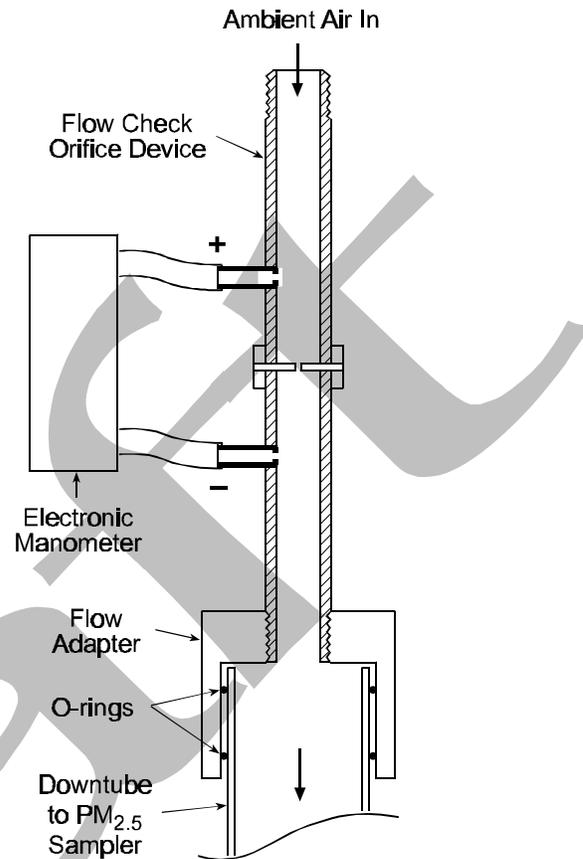


Figure 8.3. Example of installation of sampler flow-check assembly.

9.0 Maintenance

9.1 Overview

Preventive maintenance is defined as a program of positive actions aimed at preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition. It is very convenient to bring the sampler to the laboratory for major maintenance.

Records should be maintained for the maintenance schedule of each sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, and cost expenditures and should include an inventory of on-hand spare equipment for each sampler.

Recommended supplies for all maintenance activities include an alcohol-based general-purpose cleaner that leaves no residue, cotton swabs, a small soft-bristle brush, paper towels, distilled water, and miscellaneous hand tools. Additional supplies may be necessary for specific procedures, as indicated below. A compressed-air source is also recommended but not required.

All activities recommended below are in addition to those specified in the operating manual specific to the sampler and are not intended to replace those activities.

Note: Always remove the impactor and filter cassette prior to removing the inlet downtube or cleaning any of the instrument parts upstream of these items. Such activities could dislodge dirt, oil, grease, or other materials that could deposit into the impactor well or onto the sampling filter. Temporarily store the impactor and filter cassette in a clean, dry location, away from contaminating materials (dust, dirt, rain, etc.) and direct sunlight.

9.2 Five-Day Maintenance Procedures

9.2.1 Water Collector

At least every 5 sampling days, inspect the water collector bottle. Remove accumulated water, clean the interior of the bottle, inspect the seals, and replace the bottle in the holder.

9.2.2 Impactor Well

At least every 5 sampling days, disassemble and clean the impactor well (refer to Figure 9.1). If spare wells are available, this can be done in the laboratory. Separate the upper and lower portions of the well, remove the used filter from the well, wipe the two halves of the well clean with lint-free laboratory tissues or a cloth, put a new filter in the well's bottom, and add 1 ± 0.1 mL of impactor oil. Check to be sure the oil covers the filter uniformly. Reassemble the well and place it in the impactor. To reduce preparation time, it is recommended that several spare impactor wells be prepared at this time and stored in a clean dust-free container until needed.

9.3 Monthly Maintenance Procedures

9.3.1 Inlet

Figure 9.2 shows a disassembled sampling inlet. The inlet seals to the downtube with two O-rings.

If the water collector attached to the side of the inlet is glass, care should be taken during handling not to crack or break it, as the sampler will not maintain adequate vacuum during operation. The glass water collector may be either replaced with a plastic jar or wrapped with insulating tape to minimize the danger of accidental breakage.

To dismantle and clean the sampler inlet:

- Mark each assembly point of the sampler inlet with a pen or pencil to provide “match marks” during reassembly.
- Disassemble the sample inlet unit according to Figure 9.2, taking care to retain all the parts. Note: If the assembly screws appear frozen, the application of penetrating oil or commercial lubricant will make removal easier.
- Clean all interior surfaces and the bug screen with the general-purpose cleaner or compressed-air source, paying particular attention to small openings and crevices. Cotton swabs and/or a small brush are most helpful in these areas. Completely dry all components.
- Check all the O-rings for distortion, cracks, fraying, or other problems. Apply small amounts of vacuum grease or replace as necessary.
- Reassemble the unit in accordance with the previously scribed match marks. Take particular care to ensure that all O-ring seals are properly sealed and that all screws are uniformly tightened.

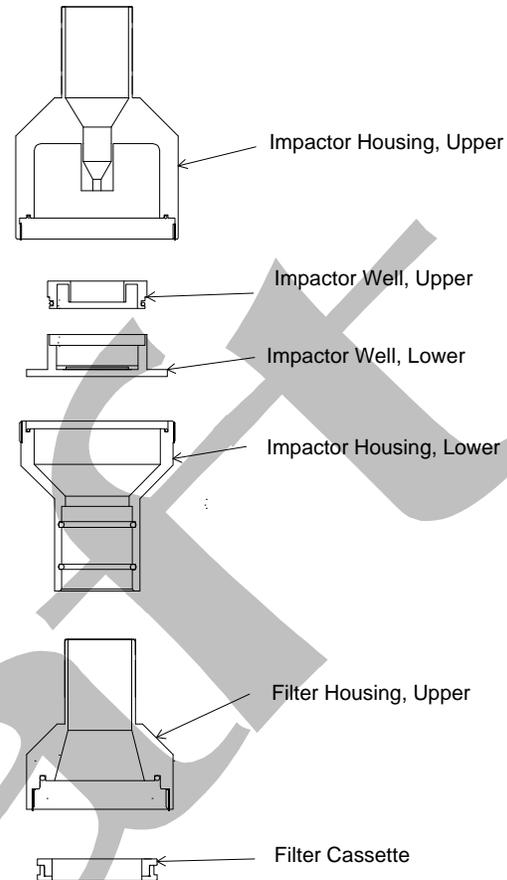


Figure 9.1. Exploded view of design of PM_{2.5} impactor well and filter holder.

9.3.2. Impactor Housing and Well

Figure 9.1 shows a typical disassembled impactor unit. With the impactor assembly open, inspect the interior of the impactor housing, both above and below the impactor well. These areas should

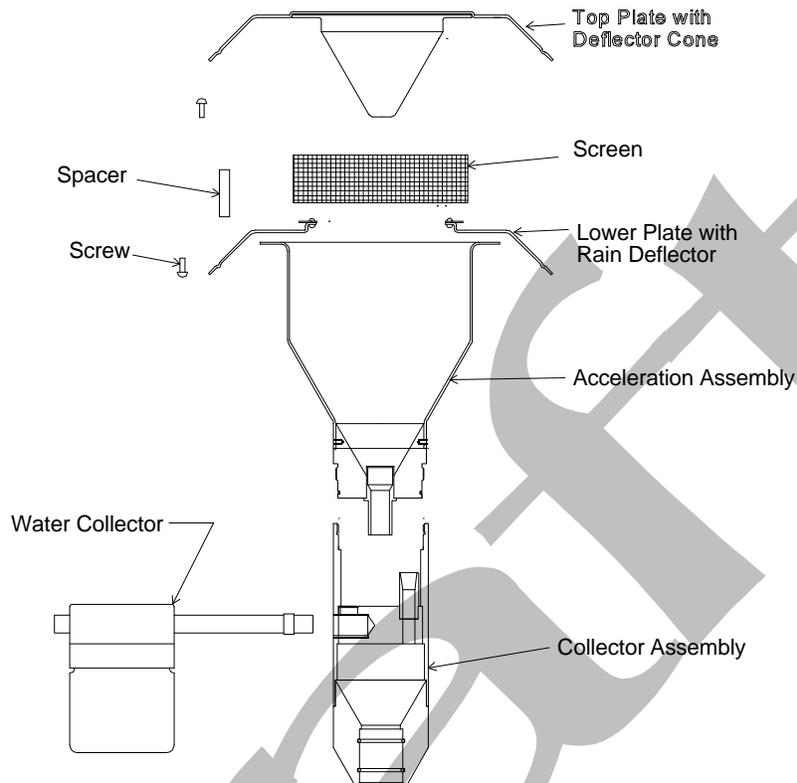


Figure 9.2. Exploded view of a PM₁₀ sampler inlet head.

be clean and dry. If necessary, clean the areas with a lint-free wipe. On a monthly basis, clean the interior of the impactor jet using a lint-free pipe cleaner or similar tool.

Check the O-rings for distortion, cracks, fraying, or other problems and replace as necessary.

9.3.3 Other

Wipe down the interior of the sampler's case to remove bugs, dirt, and/or water deposits that may have collected inside the case. This procedure may be required more frequently during summer months. Inspect the cooling air intake filter and clean it if necessary.

Check the sampler's internal clock against a timepiece known to be accurate at least to within 1 min/month. Record the value and note whether the sampler's clock has gained or lost time since the previous month's time check.

Check the integrity of the foam disks inside the metal containers used to transport the filter cassettes between the laboratory and the field. If pieces are coming loose or if the disks are discoloring, replace them.

Check the filter cassettes and the backing screens for fractures, cracks, evidence of wear, or contamination. Clean or replace as necessary.

9.4 Quarterly Maintenance

The following activities should be performed once every 3 months at the time of the regular monthly maintenance.

9.4.1 Inlet

Remove the O-rings in the aerosol inlet and condition them with vacuum grease. This will inhibit breakdown and fraying of the O-rings caused by friction on the inlet tube.

Inspect the outer and inner surfaces of the tip (closest to the sampler inlet) of the downtube, and clean any particulate deposits using alcohol or water and a soft bristle brush. Dry the downtube completely.

Inspect the water seal where the downtube enters the sampling case for cracks or other evidence of leakage. Lubricate or replace the gaskets as necessary, according to the manufacturer's operating manual.

9.4.2 Impactor

Remove the O-rings in the impactor assembly and condition them with vacuum grease.

9.4.3 Other

Examine the vacuum tubing for crimps, cracks, or breaks and replace as necessary.

Examine the tubing fittings to ensure proper seating.

Clean or replace the cooling air intake filter as required.

9.5 Other Maintenance

The pump will need to be rebuilt after a certain period of time (typically a few thousand hours of operation) depending on the manufacturer and the typical sampling conditions. Because pumps can differ substantially in design and construction, the details of rebuilding them must come from the vendor. Pump rebuilding kits are typically available from the same manufacturer from which the sampler was purchased.

Perform a leak check and recalibration of the flow system after performing any maintenance of this sort, prior to sample collection.

9.6 Refurbishment of PM_{2.5} Samplers

PM_{2.5} samplers that have been operated in the field for extended periods may require major repairs or complete refurbishment. In these cases, the manufacturer's instrument manual must be referred to before work is undertaken. A sampler that has been subject to major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

10.0 Auditing Procedures

10.1 Overview

Agencies providing PM_{2.5} monitoring data for the SLAMS program must perform QA audits and must determine and report the accuracy and precision of PM_{2.5} monitoring systems that they operate. Audits for PM_{2.5} measurement systems are to be carried out in accordance with relevant sections of 40 CFR Parts 58, 50, and 53 (EPA 1997a, 1997b, 1997c). This section of the QA handbook provides technical guidance and recommendations for conducting audits and assessments of data quality for PM_{2.5} measurement systems. True assessment of the accuracy and correctness of the PM_{2.5} measurement system can only be achieved when the audit is conducted under the following guidelines:

- Without special preparation or adjustment of the system to be audited
- By an individual with a thorough knowledge of the instrument or process being evaluated but not by the routine operator
- With accurate, calibrated, NIST-traceable transfer standards that are completely independent of those used for routine calibration and QC flow checks
- With complete documentation of audit information. This documentation includes but is not limited to types of instruments and audit transfer standards, model and serial numbers, transfer-standard traceability, calibration information, and the collected audit data.

Enhanced QA will be required in all areas relating to PM_{2.5} sampler performance. A precision assessment involves auditing each monitoring location using collocated sampler. Assessment of accuracy involves collocating an FRM sampler near the station sampler. The user agency will be required to obtain four collocated measurements per year with a reference method “audit” sampler for 25 percent of all routinely operating PM_{2.5} monitors. In addition, 25 percent of all PM_{2.5} samplers will be permanently collocated with a reference or equivalent PM_{2.5} sampler. Data from these audits and collocated samplers will be used to assess operating performance nationally and to identify reporting organizations or individual sites that have unacceptable bias or imprecision.

The specific procedures for conducting audits and evaluations of the following portions of the total PM_{2.5} measurement system are provided in these sections:

- Performance Audits (leak test, temperature, pressure, and sampler flow) (Section 10.2)
- Assessment of Accuracy and Precision (Section 10.3)
- System Audits (Section 10.4).

10.2 Performance Audits

This section presents performance audit procedures specific to reference or Class I equivalent PM_{2.5} samplers that operate on the inertial impaction principle at a sample flow rate of 16.7 L/min. Procedures for auditing samplers that operate according to other principles (e.g., open-path optical monitors) are not provided herein. 40 CFR Part 58, Appendix A (EPA 1996), requires that the flow rate accuracy and bias of individual PM_{2.5} samplers used in SLAMS monitoring networks

be assessed periodically through audits of each sampler's operational flow rate. In addition, 40 CFR Part 58, Appendix A, requires that flow rate bias for each reference and equivalent method operated by each reporting organization be assessed quarterly and annually.

Flow rate is the primary variable influencing accuracy and precision of the reference or Class I equivalent method for $PM_{2.5}$. To accurately assess the flow rate control that a sampler has achieved, it is necessary to perform the following audit checks prior to the flow rate audit itself. If scheduling permits, the site operator could conduct these checks in the presence of the auditor. These are the same checks the operator is to make every 5 days of sampler operation (refer to Section 8.3.2).

- Leak rate check
- Ambient temperature sensor audit
- Filter temperature sensor audit
- Barometric pressure sensor check.

10.2.1 Audit Preparations and Standards

Audit data sheets similar to those shown in Figures 10.1 and 10.2 should be used to document information about the equipment and the site to be audited. This information should include, but not be limited to, the sampler and audit transfer standard type, model, and serial numbers. The sampler leak check data and a check of the accuracy of the sampler's internal clock display are also included on this sheet. For automated data acquisition, a printout may be attached to this and subsequent audit data sheets. The audit data sheets shown in the figures may be modified to include additional information as necessary. In conducting the checks and audits described in the following sections, the auditor should consult the manufacturer's operations manual for sampler-specific procedures, suggestions, and precautions.

Any type of flow rate transfer device acceptable for use in the calibration of $PM_{2.5}$ samplers may be used as the audit flow rate reference standard; however, the audit standard **must** be a different device from the one used to calibrate the sampler. The audit standard must be calibrated against a standard traceable to NIST. It is acceptable for the audit and calibration standards to be individually traceable to the same NIST primary standard.

10.2.2 Leak Check

Two types of leak checks are specified for use with $PM_{2.5}$ samplers—an external leak check and an internal filter bypass leak check. The results for both of these procedures should be recorded on a form such as the one shown in Figure 10.1. Each leak check procedure is described below.

10.2.2.1 External Leak Check

The sampler components to be subjected to this leak test include all components and their interconnections in which external air leakage would or could cause an error in the sampler's measurement of the total volume of sample air that passes through the sample filter.

All $PM_{2.5}$ samplers must include external air leak test components, accessory hardware, operator interface controls, a written procedure, and all other necessary functional capability to carry out a

PM_{2.5} Sampler Audit Data Sheet
Part 1—General Information and Leak Check, Time Check

Site Location: _____ Site Designation: _____

Auditor Name and Affiliation: _____

Observer Name(s) and Affiliation(s): _____

Sampler Model: _____ Sampler ID: _____

Leak Checks: _____ Date of Test: _____

External			Internal	
Time	Indicated Pressure, mm Hg		Time	Indicated Pressure, mm Hg

Note: Repeat test or make repairs if necessary to achieve minimal leakage before proceeding with flow rate audit.

Time Check: Sampler Time _____ Reference Time _____

Comments: _____

Figure 10.1. Audit information sheet.

PM_{2.5} Sampler Audit Data Sheet
Part 2—Temperature, Pressure, and Flow-Rate Audit

Sampler Model: **IO-2001** Sampler ID: **R-102**

Pressure Audit Device Manufacturer, Model, Serial Number: **Hand-held aneroid barometer, S/N 420292**

Temperature Audit Device Manufacturer, Model, Serial Number: **Thermistor temperature probe, S/N T01562-53**

Flow Rate Audit Device Manufacturer, Model, Serial Number: **Orifice device, S/N 104, coupled to electronic digital micromanometer**

Traceability Information. Record the identifying information for NIST-traceable reference standards used to certify the audit devices:

Pressure: **Fortin (mercury column) barometer. Checked versus local NWS station**

Temperature: **ASTM 15-043A mercury-in-glass thermometer**

Flow: **soap film flowmeter primary flow device (NIST traceable)**

Temperature and Pressure Audit: _____ Date of Test: **8/13/97**

Sampler's Indicated T _a (°C)	Audit T _a (°C)	Sampler's Indicated P _a (mm Hg)	Audit P _a (mm Hg)
33.7	33.1	759	760

Flow Rate Audit: _____ Date of Test: **8/13/97**

Sampler's Indicated Flow Rate, L/min	Audit Flow Rate, L/min	Difference (Sampler - Audit), L/min	Difference ÷ Audit Flow, %
16.72	16.59	0.13	0.8%

Final Flow Rate after Audit **16.72** L/min

Notes: Attach additional sheets, if necessary.
 Show calculations on attached sheets.

Figure 10.2. Temperature, pressure, and flow-rate audit form.

leak test of the sampler at a field monitoring site without additional equipment. This capability is used to conduct the leak check during the performance audit.

Follow these steps to perform the external leakage procedure:

1. Remove the sampler inlet and install the flow rate measurement adapter supplied with the sampler (Figure 10.3). Install a leak-check filter in the sampler filter holder.
2. Close the valve on the flow rate measurement adapter and use the sampler air pump to draw a partial vacuum into the sampler, including (at least) the impactor, the filter holder assembly (filter in place), the flow measurement device, and the interconnections between these devices, of at least 55 mm Hg (75-cm water column), measured at a location downstream of the filter holder assembly.
3. Plug the flow system downstream of these components to isolate the components under vacuum from the pump, such as with a built-in valve.
4. Stop the pump.
5. Measure the trapped vacuum in the sampler with a built-in pressure-measuring device.
6. Measure the vacuum in the sampler with the built-in pressure-measuring device again at least 10 minutes after the first measurement.

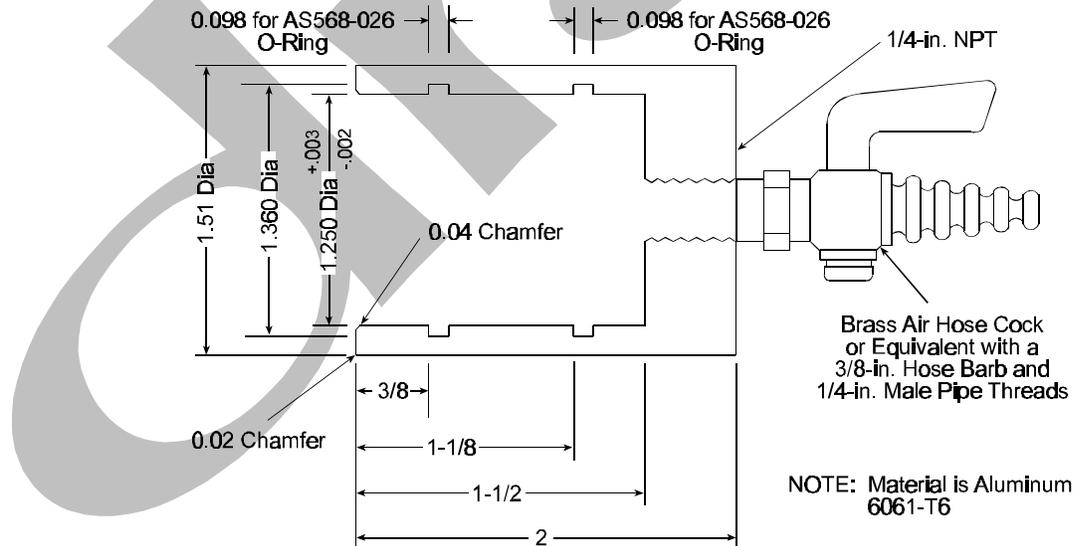


Figure 10.3. Flow adapter.

CAUTION

Following completion of the test, the adapter valve should be opened slowly to limit the flow rate of air into the sampler. An excessive air flow rate may blow oil out of the impactor well and contaminate surfaces.

7. Upon completion of the test, open the adapter valve, remove the adapter and plugs, and restore the sampler to the normal operating configuration.

To pass the external leakage test, the difference between the two pressure measurements should not be greater than the number of millimeters of Hg specified for the sampler by the manufacturer, based on the actual interior volume of the sampler, that indicates a leak of less than 80 mL/min.

Variations of the suggested technique or an alternative external leak test technique may be required for samplers whose design or configuration would make the suggested technique impossible or impractical. Therefore, a manufacturer may propose an alternative procedure to EPA under the reference or equivalency application under 40 CFR Part 53. In such cases, the manufacturer's EPA-approved leak test procedure should be followed.

10.2.2.2 Internal Filter Bypass Leak Check

The purpose of this test is to determine if any portion of the sample flow rate that leaks past the sample filter without passing through the filter is significant relative to the design flow rate for the sampler. The suggested technique for the operator or auditor to use for this leak test is as follows:

1. Carry out an external leak test as described above.
2. Install a **flow-impervious** membrane material in the filter cassette, either with or without a filter, as appropriate, which effectively prevents air flow through the filter.
3. Use the sampler air pump to draw a partial vacuum in the sampler, downstream of the filter holder assembly, of at least 55 mm Hg (75-cm water column).
4. Plug the flow system downstream of the filter holder to isolate the components under vacuum from the pump, such as with a built-in valve.
5. Stop the pump.
6. Measure the trapped vacuum in the sampler with a built-in pressure-measuring device.
7. Measure the vacuum in the sampler with the built-in pressure-measuring device again at least 10 min after the first pressure measurement.
8. Remove the flow plug and membrane, and restore the sampler to the normal operating configuration.

For successful passage of this test, the difference between the two pressure measurements should not be greater than the number of millimeters of Hg specified for the sampler by the manufacturer, based on the actual internal volume of the portion of the sampler under vacuum, that indicates a leak of less than 80 mL/min. As with the external leak check procedure described above,

variations of the suggested technique may be used, provided that they are approved as part of the manufacturer's reference or equivalency application for that sampler.

If the leak rate for either of the above procedures is excessive, note the problem on the audit data form (Figure 10.1). With the cooperation of the site operator, attempt to remedy excessive leakage before proceeding with the remainder of the audit. Check the O-rings on the flow rate adapter for nicks, wear, and excessive flattening. Also check the outside of the intake tube (the downtube) where the device seats for deep scratches or dirt that could allow leakage. Check that the filter holder is well seated. Rectify any problems, and repeat the leak check(s).

10.2.3 Temperature and Barometric Pressure Audit Procedures

Ambient temperature, filter temperature, and ambient pressure sensors on the PM_{2.5} sampler are to be audited in conjunction with the flow rate audit. Record the audit data on a form similar to the one shown in Figure 10.2.

10.2.3.1 Temperature

The recommended procedure for auditing temperature probes is to use a bath of water, oil, or other suitable liquid to provide a stable temperature. A container such as a thermos bottle or Dewar flask should be used to insulate the bath, minimizing heat transfer to and from the fluid in the flask. Temperature sensors and thermometers should never be used to stir the bath. If water is used for the bath, be sure that evaporative cooling does not cause the temperature in the bath to fall during the audit process. A minimum of three temperatures should be used for the audit. Temperatures should be approximately equally spaced and should span the temperature range expected at the site over the next 6 months. A deionized water/ice slurry can conveniently be used to achieve a temperature around 0 °C. The temperature bath liquid should be stirred to ensure that the temperature is uniform. Stirring should continue, if possible, while measurements are being made; however, do not stir while readings are being taken as this motion might damage temperature sensors or introduce noise into the readings. Allow the sensors to stabilize for several minutes at each temperature. Record the stabilized temperature reading and report the results on a data set such as the one shown in Figure 10.2. Any deviation greater than 2 °C should be reported to the QA supervisor for corrective action.

If the sensor cannot be removed for auditing, it is tempting to perform the audit by collocating the station sensor with the audit sensor and reading the temperature of the ambient air. This procedure should be avoided because the rate of temperature stabilization is much poorer in air than in a stirred liquid, and only a single temperature point can be checked. Such a procedure may be acceptable for certain routine checks of operability but should be avoided for audits.

10.2.3.2 Barometric Pressure

Ambient pressure is audited using a suitably calibrated sensor at ambient conditions. Because this is only a single-point check, additional data for comparison can optionally be obtained from a nearby National Weather Service (NWS) station. An appropriate pressure offset due to differences in elevation between the monitoring station and the NWS station must be applied. Any deviation greater than 10 mm Hg should be reported to the QA supervisor for corrective action.

10.2.4 Flow-Rate Audit Procedures

The operator or auditor must carry out the leak tests described above and correct excessive leakage before conducting the flow rate audit. The calibration relationship between the flow rate (in actual L/min) indicated by the flow rate standard and by the sampler's flow rate measurement device should be established or verified in accordance with the sampler's operation or instruction manual prior to the audit; however, no adjustments may be made to the flow rate sensors or controls before the audit is performed.

The audit of the sampler's flow rate should consist of one measurement made at the sampler's operational flow rate. The sampler's operation or instruction manual should provide specific instructions for using the instrument's flow rate readout equipment. The audit is carried out as follows:

1. Instruct the operator to install a new filter in the filter holder and energize the sampler. Filters used for flow-rate audits should not be used for sampling.
2. Verify that the flow rate is set to the designated flow rate of 16.7 L/min. (If the sampler flow-rate readout indicates some other flow rate, record this fact on the audit data sheet, and reset the flow rate to 16.7 L/min according to the manufacturer's instructions.)
3. Allow the sampler to warm up for a minimum of 5 minutes to allow the sampler's electronics to stabilize.
4. Complete the required information at the top of the flow-rate audit data sheet (Figure 10.2).
5. Remove the sampler inlet and install the flow-rate adapter device (Figure 10.3).
6. Connect the adapter to the flow-rate transfer standard outlet with flexible tubing, being careful not to crimp the tubing. If the transfer standard is electronic (such as an electronic micromanometer coupled to an orifice device), a warmup time of at least 5 minutes is recommended.
7. Recheck the flow rate indicated by the PM_{2.5} sampler's data display or other readout device. Record this value and the corresponding flow rate indicated by the audit device.
8. Record at least three sampler and audit flow rate readings of the output data directly on the audit data sheet (Figure 10.2). If electronic recording devices are used for one or more of the parameters, be sure that the clocks are synchronized. If units other than actual flow are output by either the sampler or the audit device, calculations will have to be done at a later time. See Section 10.2.5 for calculation methods. Record these results on the data sheet.
9. Calculate the overall error in flow rate in L/min and as a percentage of the total flow.

Following the measurement with the flow-rate standard, remove the adapter and reinstall the sampler inlet. Determine the sampler's operating flow rate (in L/min) with a clean filter in place. Record the final flow on the audit data sheet shown in Figure 10.2. Do not use the filter for further sampling.

10.2.5 Audit Data Calculations

10.2.5.1 Correction to Standard Conditions

PM_{2.5} flow-rate audit results should be reported at ambient conditions and should not be corrected to standard conditions (e.g., 25°, 760 torr). However, for some types of audit devices, it may be necessary to correct the indicated audit flow rates to ambient conditions. Equation (10-1) may be used to make this correction. The following correction should only be applied if the audit device reports flow rate already corrected to standard conditions (e.g., mass flowmeters). Be sure to verify the standard conditions of temperature and pressure (T_{std} and P_{std} , respectively) applicable to the audit device.

$$Q_a = Q_{std} (T_a/P_a) (P_{std}/T_{std}) \quad (10-1)$$

where

- Q_a = flow rate at actual conditions, L/min
- Q_{std} = flow rate reported at standard temperature and pressure (e.g., 25 °C, 298 K; 760 mm Hg or 101 kPa), L/min
- T_a = ambient temperature, K
- P_a = ambient barometric pressure, mm Hg or kPa
- P_{std} , T_{std} = standard barometric pressure and temperature, respectively.

10.2.5.2 Audit Percentage Difference Calculation

Determine the percentage difference between the sampler-indicated flow rates and the audit-measured flow rates using Equation (10-2). Record the result on the audit data sheet. Any deviation greater than ± 2 percent may require recalibration.

$$\% \text{ difference} = 100 \times (Q_a(\text{sampler}) - Q_a(\text{audit})) / Q_a(\text{audit}) . \quad (10-2)$$

10.2.6 Balance Audits

A performance audit of the microbalances used to weigh PM_{2.5} filters will require the use of ASTM Class I standard weights. Because microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the performance evaluation of the filter-weighing process be done in cooperation with the laboratory personnel. The person normally performing the weighings for PM_{2.5} monitoring should assist the auditor. Prepare a form similar to the audit information sheet shown in Figure 10.1 to record observations and data.

1. Review the maintenance and calibration log for each balance. Routine balance maintenance and calibrations must be performed by the manufacturer's service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed 1 year.
2. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:
 - Zero and working mass reference standards checks after approximately every 10 filter weighings
 - Laboratory blank filter weighings every day of balance operation

- Field blank filter weighings for each scheduled sampling day
- Periodic duplicate filter weighings.

If any QC checks were out of limits, note what action was taken.

3. Observe the balance operator's technique and review the laboratory weighing procedure for determining both the tare and gross weights of the sampling filters. In addition, take the following steps:
 - Select randomly and have the balance operator reweigh 4 equilibrated filters out of every group of 50 or less from the most recent group of filters weighed. For groups of 50 to 100, reweigh 7 from each group.
 - Record the original values and the audit weights on the audit form. Calculate the weight difference for each filter as follows:

$$\text{Difference} = \text{Original weight (mg)} - \text{Audit weight (mg)} .$$

For unexposed filters, the difference should be less than $\pm 15 \mu\text{g}$ (0.015 mg). For exposed filters, the potential loss of volatile particles prevents establishment of meaningful acceptance or rejection limits. Forward the audit data to the laboratory QA supervisor for review.

10.3 Assessment of Accuracy and Precision

10.3.1 Assessment of Accuracy

Because the size and volatility of the particles making up ambient particulate matter vary over a wide range and the mass concentration of particles varies with particle size, it is difficult to define the accuracy of $\text{PM}_{2.5}$ measurements in an absolute sense. The accuracy of $\text{PM}_{2.5}$ measurements is therefore defined in a relative sense, referenced to measurements provided by the FRM. Accuracy is defined as the degree of agreement between a subject field $\text{PM}_{2.5}$ sampler (the primary sampler) and a collocated FRM audit sampler operating simultaneously at the monitoring site. Audit results will include both random (precision) and systematic (bias) errors.

The schedule and procedure for performing collocated measurements with the FRM sampler are provided in 40 CFR Part 58, Appendix A. The results from the primary sampler and the duplicate FRM sampler are used to calculate the accuracy of the primary sampler on a quarterly basis, the bias of the primary sampler on an annual basis, and the bias of a single reporting organization on an annual basis.

10.3.2 Assessment of Precision

Collocated sampler results, where the duplicate sampler is not a reference method sampler but a sampler of the same designated method as the primary sampler, are used to assess measurement system precision according to the schedule and procedure specified in 40 CFR Part 58, Appendix A. These collocated sampler measurements are used to calculate quarterly and annual precision estimates for each primary sampler and for each designated method employed by each reporting organization.

An objective of 10 percent coefficient of variation or better has been established for the

operational precision of PM_{2.5} monitoring data.

10.3.3 Procedure for Collocated Measurements

The FRM or audit sampler must be a sampler associated with a PM_{2.5} method that has been designated by the EPA as a reference method under 40 CFR Part 53. In addition, the sampler used for collocation should be inspected frequently for proper operation, and the calibration of its flow rate, temperature, and pressure measurement systems should be checked and verified at least every 3 months.

Audits using collocated samplers are performed by installing and operating an audit or FRM sampler concurrently with each routinely operating PM_{2.5} sampler. The station and audit samplers must be located at least 2 m but no more than 4 m apart, and their inlets should be at the same height above ground and within 1 m of each other as measured in the vertical direction. The samplers should be calibrated and operated, and the sample filters analyzed, as described in the sampler's operation or instruction manual and in general accordance with the guidance in this handbook. The station sampler must be operated during the audit as closely as possible to the way it operates during normal sampling or monitoring. Audits should be conducted during a regular sample period or during normal monitoring whenever possible. The station sampler should not receive any special or nonscheduled service prior to the audit, with the exception that a normal leak test should be carried out before each audit for all samplers.

The concentrations determined from the station sampler and from the audit sampler should be reported to EPA as directed in 40 CFR Part 58, Appendix A, Section 4. It should be particularly noted that all collocated audit measurements must be reported, even those that might be considered invalid because of identified malfunctions or other problems that occurred during or following the sample collection period.

10.4 System Audits

System audits are intended to assess the entire measurement system and data collection activities. These activities include the following:

- Initial equilibration, weighing, and transportation of the filters to the sampler
- Site selection criteria assessment
- Equipment installation
- Site security
- Equipment maintenance
- Calibration procedures
- Handling and placement of the filters
- Proper operation of the sampler and sample collection
- Removal, handling, and transportation of the filter from the sampler to the laboratory
- Weighing, storage, and archival of the sampled filter
- Data analysis and reporting.

EPA specifies that QA and QC programs follow the requirements for QA project plans (QAPPs) contained in the EPA document *Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5 (EPA 1994). Auditors should develop a checklist

based on these guidelines and on the specific information regarding PM_{2.5} monitoring in this QA handbook.

Effective system auditing requires the auditor to observe the auditee's entire sample acquisition and analysis system and the associated data processing operations. A major portion of the system audit should be performed while the auditor is at the sampling site conducting the performance audit described previously. Other locations where the system auditor should observe operations include the laboratory, the shipping and receiving area where field samples are received and logged in, the sample archival area, and the data processing areas.

Data about specific samples obtained during the on-site performance audits may be useful during the system audit. Tracking specific samples and the associated data completely through the system (sample acquisition, shipping, receiving, logging in, weighing, archiving, data processing, and reporting) ensures that every significant operation is examined. This approach also allows the auditor to look at the "interfaces" between different operations—the interactions between field and laboratory tasks—and not just the individual tasks in isolation. Detailed and summary reports and data submitted to EPA databases (e.g., AIRS) for the audited period should be reviewed as a followup to the audit, if the timing of these reports permits.

Effective preparation for a system audit requires developing a checklist similar to the one shown in Figure 10.4. Auditors are encouraged to develop their own checklists based on their experience and as dictated by the individual audit situation.

Checklist for PM_{2.5} Auditing				
Site Location _____		Site Designation _____		
Auditor Name and Affiliation _____				
Observer(s) Name and Affiliation _____				
Audit Question	Y	N	N/A	Comment
On Site:				
• Are logbooks and required data sheets filled in promptly, clearly, and completely?				
• Are filters handled with the necessary care and finesse to avoid contamination and/or loss of material? Does the operator keep the filter-handling area neat and clean?				
• Is the equipment sited properly? Are there any changes at the site that might compromise original siting criteria (e.g., fast-growing trees or shrubs, new construction)?				
• Does the equipment appear to be well maintained and free of dirt and debris, bird/animal/insect nests, excessive rust and corrosion, etc.?				
• Are the walkways to the station and equipment kept free of tall grass, weeds, and debris?				
• Is the station shelter (if any) clean and in good repair?				

(continued)

Figure 10.4. System audit questionnaire.

Audit Question	Y	N	N/A	Comment										
Sampler Calibration and Maintenance:														
<ul style="list-style-type: none"> Does the flow rate standard used for routine flow rate calibration have its own certification traceable to a NIST primary standard for volume of flow rate? 														
<ul style="list-style-type: none"> Is the calibration relationship for the flow rate standard (e.g., an equation, curve, or family of curves relating actual flow rate [Q_a] to the flow rate indicator reading) accurate to within 2 percent over the expected range of ambient temperatures and pressures at which the flow rate standard may be used? Verify by inspecting the original calibration data and calculating the error in the calibration relationship for each calibration point used to generate the relationship. 														
<ul style="list-style-type: none"> Is the flow rate standard used for routine sampler calibration/verification recalibrated or reverified against a NIST-traceable standard at least annually? 														
Balance Maintenance and Weighing Procedures														
<ul style="list-style-type: none"> Is the filter preparation and weighing area neat and clean? 														
<ul style="list-style-type: none"> Are logbooks kept up to date and properly filled in? 														
<ul style="list-style-type: none"> Is the balance on a service agreement for regular professional maintenance? If not, is there someone in the organization with the appropriate training to service the balance? 														
<ul style="list-style-type: none"> Does the analytical balance used to weight filters have a readability of ±1 µg? 														
<ul style="list-style-type: none"> Is there a formal logbook or file for balance maintenance? Are entries current? 														
<ul style="list-style-type: none"> Are regular (e.g., daily, when in use) calibration checks made and recorded? 														
<ul style="list-style-type: none"> Are filters conditioned immediately before both the pre- and postsampling weighings? The following conditions are specified in 40 CFR Part 58, Appendix L: 														
<table border="1"> <tr> <td>Mean temperature:</td> <td>20–23 °C.</td> </tr> <tr> <td>Temperature control:</td> <td>±2 °C over 24 hours.</td> </tr> <tr> <td>Average humidity:</td> <td>30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling.</td> </tr> <tr> <td>Humidity control:</td> <td>±5 percent RH over 24 hours.</td> </tr> <tr> <td>Conditioning time:</td> <td>Not less than 24 hours.</td> </tr> </table>					Mean temperature:	20–23 °C.	Temperature control:	±2 °C over 24 hours.	Average humidity:	30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling.	Humidity control:	±5 percent RH over 24 hours.	Conditioning time:	Not less than 24 hours.
Mean temperature:	20–23 °C.													
Temperature control:	±2 °C over 24 hours.													
Average humidity:	30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling.													
Humidity control:	±5 percent RH over 24 hours.													
Conditioning time:	Not less than 24 hours.													
(continued)														

Figure 10.4 (continued)

Audit Question	Y	N	N/A	Comment
Balance Maintenance and Weighing Procedures (continued):				
• Are new filters placed in the conditioning environment immediately upon arrival and stored there until the presampling weighing?				
• Is the analytical balance located in the same controlled environment in which the filters are conditioned?				
• Are the filters weighed immediately following the conditioning period without intermediate or transient exposure to other conditions or environments?				
• Are filters conditioned at the same environmental conditions before both the pre- and postsampling weighings?				
• Are both the pre- and postsampling weighings carried out on the same analytical balance, using an effective technique to neutralize static charges on the filter?				
• Are both weighings carried out by the same analyst? (If not, have results of the different analysts been compared statistically?)				
• Are presampling weighings done within 30 days of the sampling periods in which filters are to be exposed? (Review logbook to look for evidence of filters that exceed their time span.)				
• If filters are stored at ambient temperature, are the post-sampling conditions and weighings completed within 240 hours (10 days) after the end of the sample period?				
• If filters are stored at 4 °C or less during the entire time between retrieval from the sampler and start of the conditioning, are the postsampling conditioning and weighings completed within 30 days after the end of the sample period?				
• Are new field blank filters weighed along with the pre-sampling (tare) weighing of each lot of PM _{2.5} filters?				
• Are QC field blank filters routinely used, observing the following handling steps: transport to the sampling site, installation in the sampler, retrieval from the sampler (without sampling), and reweighing?				
• Are QC laboratory blank filters weighed along with the pre-sampling (tare) weighing of each set of PM _{2.5} filters and reweighed when the exposed filters are received from the field? (These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a QC check.)				
• Was the balance calibrated as specified by the manufacturer at installation and recalibrated immediately prior to each weighing session?				

Figure 10.4 (continued)

11.0 Calculations, Validations, and Reporting of PM_{2.5} Monitoring Data

11.1 Overview

This section presents the calculations required to compute and report ambient PM_{2.5} concentrations. A summary of all calculation formulas is given in Table 11-1. Associated symbols are listed in Table 11-2.

The end result of these calculations will be the integrated (average) PM_{2.5} concentration during the sampling period, expressed in units of µg/m³. To obtain this concentration, it is necessary to

**TABLE 11-1. FORMULAS FOR CALCULATIONS ASSOCIATED WITH
PM_{2.5} MONITORING**

Calculation	Formula	Equation No.	Section
Alternative calculation of total sample volume, used only if V _a is not available directly from the sampler	$V_a = Q_{ave} \times t \times 10^{-3}$	1	11.2.1
Determination of PM _{2.5} mass collected on filter (net weight)	$M_{2.5} = (M_f - M_i) \times 10^3$	2	11.2.2
Determination of PM _{2.5} mass concentration	$PM_{2.5} = M_{2.5}/V_a$	3	11.2.3

**TABLE 11-2. SYMBOLS USED FOR CALCULATIONS ASSOCIATED WITH
PM_{2.5} MONITORING**

Symbol	Description	Units
M _{2.5}	Total mass of PM _{2.5} particles	µg
PM _{2.5}	Concentration of PM _{2.5} particles	µg/m ³
Q _{ave}	Average sample flow rate	L/min
t	Elapsed sample collection time	min
V _a	Total sampled volume (at ambient temperature and pressure)	m ³
M _i	Initial equilibrated mass of filter (before sample collection)	mg
M _f	Final equilibrated mass of filter (after sample collection)	mg

determine the mass of particles collected by the filter and the total volume of air that was sampled. Unlike many other pollutant measurements, the volumes used to calculate the PM_{2.5} concentration **must** always be expressed at the **actual** ambient temperature and pressure used to acquire the sample (40 CFR Part 50.3, as amended 6/28/97). This process represents a change from the convention used to report PM measurements prior to June 1997, in which volumes were adjusted to “EPA-standard” conditions, defined as 25 °C (298 K) and 760 mm Hg (101 kPa).

Accurate measurement of the sampler’s flow rate is needed both to determine the total sampled volume and to ensure that the particles trapped by the filter are in the correct particle size range. Reference samplers and Class I equivalent samplers separate PM_{2.5} particles by inertia. Obtaining the correct particle size range by inertial separation requires that the correct air velocity be maintained in the sampler’s size separator system (impactor). This velocity will be correct when the specified design flow rate is maintained. The design flow rate is specified at the sampler’s actual ambient temperature and pressure (i.e., not corrected to standard conditions).

11.2 Calculations

11.2.1 Sample Volume Calculations

Both reference and equivalent method samplers are required to provide measurements of the total volume of air sampled (V_a), in m³ at the actual ambient temperatures and pressures during sampling (40 CFR Part 50, Appendix L, paragraph 7.4.5.2). If the sampler’s flow measurement system is properly calibrated, V_a should be accurate, and no further sample volume calculations are required.

Note that in the event the total sample volume measurement from the sampler is not available, the total sample volume may be calculated by multiplying the average flow rate, in actual m³/min, by the elapsed sample collection time in minutes. Both of these measurements are required to be provided by reference and equivalent method samplers. Use the following formula only if V_a is not available directly from the sampler:

$$V_a = Q_{ave} \times t \times 10^{-3} \quad (11-1)$$

where

- V_a = total sample volume, actual m³
- Q_{ave} = average sample flow rate over the sample collection period, L/min
- t = total elapsed sample collection time, min
- 10^{-3} = units conversion (m³/L).

For example, a sampler with an average flow rate of 16.7 L/min (Q_{ave}) for a 1,410-min (23.5-hour) sampling period (t) would have a total sample volume (V_a) of 23.5 m³.

11.2.2 Net PM_{2.5} Mass Calculation

The mass of particulate matter collected on the filter during the sampling period is determined by subtracting the initial (tare) mass of each filter from the final mass of the filter, as

$$M_{2.5} = (M_f - M_i) \times 10^3 \quad (11-2)$$

where

- $M_{2.5}$ = total mass of PM_{2.5} collected during the sampling period, μg
- M_f = final mass of the equilibrated filter after sample collection, mg
- M_i = initial (tare) mass of the equilibrated filter before sample collection, mg
- 10^3 = units conversion ($\mu\text{g}/\text{mg}$).

For example, a filter that weighed 139.293 mg before sampling (M_i) and 139.727 mg after sampling (M_f) would have a PM_{2.5} mass ($M_{2.5}$) of 434 μg .

11.2.3 PM_{2.5} Concentration Calculation

Each PM_{2.5} mass concentration measurement is calculated by dividing the total mass of PM_{2.5} (Equation 11-2) collected during the sampling period ($M_{2.5}$) by the total volume of air sampled (V_a) (taken directly from the sampler readout display or calculated from Equation 11-1), as

$$\text{PM}_{2.5} = M_{2.5} / V_a \quad (11-3)$$

For example, a sample with a mass ($M_{2.5}$) of 434 μg collected from a total sample volume (V_a) of 23.5 m^3 calculates to be a PM_{2.5} concentration ($\text{PM}_{2.5}$) of 18.5 $\mu\text{g}/\text{m}^3$.

11.3 Calculation Validation

Calculation validation ensures that PM_{2.5} measurements meet the program's QA goals. The validation procedure not only checks computations, but it also aids in detecting those PM_{2.5} concentration measurements with extremely high or low values that may indicate questionable data. The steps of the calculation validation procedure are as follows:

1. Gather the following data for each sample in the group of samples to be reported:
 - Total sample volume (V_a) in m^3
 - Tare (M_i) and final (M_f) masses of the filter in mg
 - Net ($M_{2.5}$) filter mass in μg .
2. Recompute (Sections 11.2.2 and 11.2.3) the total mass concentration of PM_{2.5} for a minimum of 7 samples or 7 samples per 100 samples in the reporting group, whichever is greater. The recomputed samples must include a minimum of four samples from each production lot of filters used. This suggested initial validation ratio may be altered, based on experience and data quality. The validation ratio may be decreased somewhat if past experience indicates that data are of good quality. However, the ratio should be increased if data are found to be of poor quality. It is more important to ensure that the validation check is representative of

the various conditions that may influence data quality than to adhere to a fixed validation ratio. If any recalculated concentration differs from the original calculation, then values for all measurements in the group (i.e., not just those selected for recomputation) should be recalculated.

3. Review all total mass concentration values. Note those that appear excessively high or low and investigate them. Recompute the PM_{2.5} concentrations (Sections 11.2.2 and 11.2.3) for such samples. Compare the validated PM_{2.5} concentration to the originally reported value. Correct any errors that are found, initial them, and indicate the date of correction.
4. If all mass concentration computations appear correct and one or more questionably high or low values still exist, review all alternative data (i.e., elapsed sample time, average actual total flow rate, total PM_{2.5} mass, and any operator notes) for verification of the correctness of the PM_{2.5} concentration computation.

11.4 Data Reporting

The primary standards for particulate matter in ambient air are based on the measured mass concentration of PM_{2.5}. Information on reporting and interpretation of PM_{2.5} data with respect to the attainment of these standards is covered in 40 CFR Part 50, Appendix K.

12.0 Data and Records Management

12.1 Introduction

It is important to keep good records in any air pollution measurement program. This is particularly important for measurements of PM_{2.5} by reference or Class I equivalent methods because these are manual methods that involve the transfer and handling of samples and data by several persons. Automated methods, on the other hand, present the final, averaged data with much less human intervention and thus limit the possibilities of transcription errors and misplacement of data.

Keeping good records and managing the data obtained under PM_{2.5} regulations is especially important because this is a newly designated criteria pollutant and the samplers and methodology for its determination are new to most users. Thus, records of knowledge gained in the initial stage of network operation will be of great help in improving and refining all operational aspects of PM_{2.5} determinations.

Section 14 of the EPA *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods, Part I*, gives guidance on data acquisition and information management (EPA 1997). Although it emphasizes automated data acquisition, much of the discussion is also applicable to manual methods. There are several reasons for maintaining complete, orderly records and properly managing data. Records can

- Provide information on mechanical problems that occur and document how the problems were corrected
- Provide a history of warranty repairs
- Provide a history of in-house repairs and preventive maintenance servicing
- Document date and site placement details for the primary and collocated samplers as well as the characteristics of the surrounding land areas, sources, and other features
- Be a useful source of information at the time of the annual network review to show proper sampler installation and operation, performance of QC and QA checks, traceability of equipment and standards, and proof that all systems were kept in control (use of control charts)
- Provide evidence to support the quality of PM_{2.5} data submitted to regional and national databases.

12.2 Methodology for Data and Records Management

A systematic approach to maintaining records and managing the data for all reported ambient air pollutant concentrations will usually involve the following considerations:

- **Personnel**—One or more persons should be identified within the reporting organization to be responsible for maintaining the records and preparing and submitting the required data products. Education, training, and experience are requirements for the position. SOPs will also be required.

- **Quality assurance**—An individual or group should be assigned to prepare SOPs, to conduct internal systems and performance audits, and to review the data before submission.
- **Facilities and equipment**—A designated storage location for paper records and computer data files should be established. Locking file cabinets is suggested. A backup copy of data files should be kept in another location for safety. Computers and data entry stations, as well as connections to centralized databases (such as AIRS), are also required.

12.3 PM_{2.5} Records to Create and Retain

A QAPP and SOPs for various aspects of the PM_{2.5} sampling program should be prepared. Much of the required information can be drawn from that given in the applicable sections of the *Code of Federal Regulations*, the sampler's instruction manual, and this document.

12.3.1 Sampler Siting and Maintenance Records

Documentation regarding siting and maintenance of the PM_{2.5} samplers should be retained in the files. These documents may include the following:

- Site selection criteria checklist; photographs or color slides of the site in the eight cardinal directions with the PM_{2.5} sampler in the center foreground; site sketch; U.S. Geological Survey (USGS) topographical map of the area; and a copy of the site documentation records that were submitted to the AIRS database when the site was first established and any updates made since that time.
- Procurement log for field equipment (see Figure 4.1). Notes on acceptance/rejection tests (see Table 4-1).
- Warranty and maintenance records for each PM_{2.5} sampler. It is recommended that a dated maintenance checklist be established for each sampler and that separate maintenance logbooks be used for the PM_{2.5} samplers and the laboratory microbalance(s).
- Manufacturer-supplied calibration and traceability records for the thermometers, flow rate measuring devices, and pressure sensors used for calibrating, checking, or auditing PM_{2.5} samplers. Maintain similar records for calibrations and traceability studies made in the reporting organization's laboratory or elsewhere.

12.3.2 Analytical Laboratory Installation Records

Records to keep regarding the physical setup of the analytical laboratory include the following:

- Equipment inventory (microbalances, conditioning chambers, antistatic devices, calibration and check weights, etc.).
- Procurement log (see Figure 4.1). Notes on acceptance/rejection tests (see Table 4-1).
- Records (e.g., control charts) that demonstrate that the temperature and RH within the weighing laboratory and conditioning chambers are being held within specified limits.
- Microbalance logbook.

12.3.3 Field Sampling Operation Records

PM_{2.5} sampling operations generate a number of paper and computerized records that need to be reviewed, reported, and filed. Starting with the calibration of the sampler's sensors, these records will include at least the following items:

- Sampler calibration logbooks or data sheets for temperature, pressure, and flow rate. Also include documentation of the devices used to calibrate the sampler(s) and these devices' traceability records or references to the traceability records. A separate section of the logbook should be used for each sampler in the network. Enter results of the sampling procedure checks (see Table 8-1) which include notes on filter inspection, and checks of flow rate and leaks.
- PM_{2.5} Sampler Run Data Sheet (see Figure 8.1). It is suggested that the run data sheet be prepared in duplicate. One way to do this is to use carbonless paper. The original of the data sheet should accompany the filter sample to the laboratory, and the copy should be retained in the field site records files.
- PM_{2.5} Sampler Flow Check Data Sheet (see Figure 8.2).
- Computer disks and printouts of data downloaded from the data port of each PM_{2.5} sampler.

12.3.4 Weighing Laboratory Operation Records

Data records from activities in the weighing laboratory are expected to include at least the following items:

- Records of temperature and humidity control in the laboratory and in the filter conditioning environment (if the latter is different from the laboratory). Use of control charts is recommended.
- Laboratory data form (see Figure 7.2).
- Laboratory internal QC log (see Figure 7.1).
- Results of microbalance calibrations and servicing. This information can be recorded in the microbalance logbook.
- Results of filter integrity checks and determinations of the conditioning periods required for various filter batches (refer to Sections 7.5 and 7.6).
- The completed PM_{2.5} Sampler Run Data Sheet (see Figure 8.1).
- Records of sample numbers (identifications) and locations of archived PM_{2.5} samples.

12.3.5 QA Procedures Records

QA system and performance audits require complete documentation as well. Records of audits may be similar to the following items:

- Audit information sheet (see Figure 10.1)
- Temperature, pressure, and flow rate audit data sheet (see Figure 10.2)
- Systems audit questionnaire (see Figure 10.4).

Accuracy and precision audit results are based on comparing the data from the primary sampler (the one used regularly at the site) to data from collocated samplers. These data are reported to the EPA Regional Office and to the AIRS data system as noted in Sections 11.0 and 12.4.

12.4 Data Reporting Requirements

The EPA Regional Office and the AIRS database system require that the following information be reported on a quarterly (generally, calendar-quarter) basis:

- Siting documentation (upon installation and any changes thereafter)
- PM_{2.5} concentration data, µg/m³ (calculated on the PM_{2.5} Sampler Run Data Sheet and entered electronically into the AIRS database system)
- Sampler-provided information (refer to Table 12-1 [40 CFR Part 50, Appendix L, Table L-1]). This information is retrieved from the data port of each sampler. It is generally downloaded in the field to a laptop computer and later processed in the laboratory or office.
- Features can be incorporated into the processing program to detect and correct typographical errors, such as out-of-sequence dates for start and end times, extremely high (or low) filter weights, flow rates, and temperatures.

TABLE 12-1. SUMMARY OF INFORMATION TO BE PROVIDED BY SAMPLER

Information to be provided	Appendix L section reference	Availability				Format	
		Anytime ^a	End of period ^b	Visual display ^c	Data output ^d	Digital reading ^e	Units
Flow rate, 30-second maximum interval	7.4.5.1	✓	—	✓	*	XX.X	L/min
Flow rate, average for the sample period	7.4.5.2	*	✓	*	✓	XX.X	L/min
Flow rate, CV, for the sample period	7.4.5.2	*	✓	*	✓●	XX.X	%
Flow rate, 5-min average out of spec. (FLAG) ^f	7.4.5.2	✓	✓	✓	✓●	On/Off	
Sample volume, total	7.4.5.2	*	✓	✓	✓●	XX.X	m ³
Temperature, ambient, 30-second interval	7.4.8	✓	—	✓	—	XX.X	°C
Temperature, ambient, min., max., average for the sample period	7.4.8	*	✓	✓	✓●	XX.X	°C
Barometric pressure, ambient, 30-second interval	7.4.9	✓	—	✓	—	XXX	mm Hg

(continued)

TABLE 12-1 (continued)

Information to be provided	Appendix L section reference	Availability				Format	
		Anytime ^a	End of period ^b	Visual display ^c	Data output ^d	Digital reading ^e	Units
Barometric pressure, ambient, min., max., average for the sample period	7.4.9	*	✓	✓	✓●	XXX	mm Hg
Filter temperature, 30-second interval	7.4.11	✓	—	✓	—	XX.X	°C
Filter temperature, differential, 30-second interval, out of spec. (FLAG) ^f	7.4.11	*	✓	✓	✓●	On/Off	
Filter temperature, maximum differential from ambient, date, time of occurrence	7.4.11	*	*	*	*	X.X, YY/MM/DD HH:mm	°C, Yr/Mo/ Day Hr min
Date and time	7.4.12	✓	—	✓	—	YY/MM/DD HH:mm	Yr/Mo/ Day Hr min
Sample start and stop time settings	7.4.12	✓	✓	✓	✓	YY/MM/DD HH:mm	Yr/Mo/ Day Hr min
Sample period start time	7.4.12	—	✓	✓	✓●	YYYY/MMM /DD HH:mm	Yr/Mo/ Day Hr min
Elapsed sample time	7.4.13	*	✓	✓	✓●	HH:mm	Hr min
Elapsed sample time out of spec. (FLAG) ^f	7.4.13	—	✓	✓	✓●	On/Off	
Power interruptions > 1 min, start time of first 10	7.4.15.5	*	✓	*	✓	1HH:mm, 2HH:mm, etc.	Hr min
User-entered information, such as sampler and site identification	7.4.16	✓	✓	✓	✓●	As entered	

✓ Provision of this information is required.

* Provision of this information is optional. If information related to the entire sample period is optionally provided prior to the end of the sample period, the value provided should be the value calculated for the portion of the sampler period completed up to the time the information is provided.

● Indicates that this information is also required to be provided to the AIRS data bank.

^a Information is required to be available to the operator at any time the sampler is operating, whether sampling or not.

^b Information relates to the entire sampler period and must be provided following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

^c Information shall be available to the operator visually.

^d Information is to be available as digital data at the sampler's data output port following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

^e Digital readings, both visual and data output, shall have no less than the number of significant digits and resolution specified.

^f Flag warnings may be displayed to the operator by a single-flag indicator or each flag may be displayed individually. Only a set (on) flag warning must be indicated; an off (unset) flag may be indicated by the absence of a flag warning. Sampler users should refer to Section 10.12 of Appendix L regarding the validity of samples for which the sampler provided an associated flag warning.

13.0 Assessment of Measurement Uncertainty for Monitoring Data

13.1 Overview

SLAMS reporting organizations are required to assess the measurement uncertainty of their PM_{2.5} monitoring data (EPA 1997). Three different procedures are used in this assessment:

- **Flow rate audit**—The accuracy of the PM_{2.5} sampler's flow rate is assessed by performing a flow rate audit as described in Section 10.2.4. The goal for accuracy of the sampler flow rate is to be within ± 2 percent of the value indicated by the audit device.
- **Bias**—PM_{2.5} measurement bias is assessed by conducting an FRM collected sampler audit under the National Performance Audit Program (NPAP). The bias goal is to be between -10 percent and +10 percent (i.e., to have the measured value within ± 10 percent of the audit value).
- **Precision**—PM_{2.5} measurement precision is assessed by collocating (i.e., locating a second, duplicate, sampler alongside the primary sampler used for measurement) samplers and reporting the results of both the measurement sampler and the collocated sampler. The precision goal is to have a coefficient of variation (CV) of less than 10 percent.

These procedures are very similar to those used in the data quality assessment of PM₁₀ monitoring data. The results of these procedures are used to screen individual samplers for bias or excessive imprecision. EPA also uses these results to assess measurement uncertainty for each reporting organization. The results from all SLAMS reporting organizations are used by EPA to assess the measurement uncertainty of data from each PM_{2.5} reference or equivalent method on a national basis.

13.2 Flow Rate Audits

Flow rate audits consist of measuring the sampler's normal operating flow rate with a flow rate transfer standard. This procedure is described in Section 10.2.4. Manual PM_{2.5} sampler methods must be audited each calendar quarter. Audits should be scheduled so as to avoid interference with regularly scheduled sampling periods. Automated samplers are required to have a one-point flow check (performed similarly to the flow check audit, Section 10.2.4) at least every 2 weeks. Where possible, these flow checks should be performed at randomly selected times with respect to time of day and day of week.

Results of the flow rate audit to be reported to EPA are as follows:

- The audit (true) flow rate as determined using the transfer standard
- The corresponding flow rate as measured by the sampler. This rate should be that used by the sampler to calculate its reported total sampling volume and average flow rate. This information is typically obtained from the sampler's flow rate display or from its data system.

The sampler's flow rate accuracy (A) should be within ± 2 percent of the audit value. The sampler's flow rate accuracy (A) is calculated as follows:

$$A(\%) = \frac{Q_{\text{Sampler}} - Q_{\text{Audit}}}{Q_{\text{Audit}}} \times 100 \quad (1)$$

where

- A = flow-rate accuracy (percent)
- Q_{Sampler} = flow rate as measured by sampler (L/min)
- Q_{Audit} = flow rate as measured by the flow rate transfer standard (L/min).

13.3 Bias Assessment

Sampler bias is assessed from the results of an FRM audit (Section 10.3) performed under the NPAP program (Hunike 1993). The goal for acceptable bias is between -10 percent and +10 percent (i.e., the reporting primary sampler's concentration should agree to within ± 10 percent of the FRM audit sampler's concentration). Minimum FRM audit requirements for SLAMS reporting organizations include the following:

- At least one sampler must be audited annually.
- At least 25 percent of each reference and each EPA-designated equivalent method must be audited each year (i.e., in most cases, 25 percent of each make and model of sampler will need to be audited). This percentage includes collocated sites, even those collocated with FRM instruments. Thus, if a reporting organization has three different makes of samplers, each with a different equivalency designation, the reporting organization will have to audit 25 percent of each make of sampler. If a fractional number of samplers is required, values of 0.5 or greater must be rounded upward to the nearest whole number. For example, a reporting organization with 10 samplers of a given type must audit at least 3 of those samplers.
- Audits must occur at least four times a year.
- All samplers must be audited at least once every 4 years.

In addition to these requirements, areas that exceed the NAAQS for $PM_{2.5}$ should place special emphasis on those sites with the highest $PM_{2.5}$ concentrations as follows:

- Reporting organizations with sites reporting $PM_{2.5}$ concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate):
 - 80 percent of FRM audits should be performed at those sites reporting $PM_{2.5}$ concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate).
 - The remaining 20 percent of FRM audits should be performed at sites reporting concentrations less than 90 percent of the NAAQS (annual or 24-hour, as appropriate).
- Reporting organizations **without** sites reporting $PM_{2.5}$ concentrations exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate):
 - 60 percent of FRM audits should be performed at sites that rank in the top 25 percent of the highest sites for $PM_{2.5}$ concentrations (annual or 24-hour, as appropriate).

- The remaining 40 percent of FRM audits should be performed at the remaining 75 percent of sites.

Additional details on NPAP audits can be obtained from the appropriate EPA Regional QA Coordinator or from the NPAP Coordinator at the following address:

NPAP Coordinator
Emissions Measurement and Analysis Division (MD-14)
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

The individual sampler and audit measurements must be reported to EPA, which uses them to calculate the single sampler bias (B) and the quarterly average bias (\bar{B}) for a reporting organization. Reporting organizations may also want to calculate these parameters for their QA use. The single sampler bias (B) may be calculated as follows:

$$B(\%) = \frac{PM_{2.5\text{Sampler}} - PM_{2.5\text{Audit}}}{M_{\text{Audit}}} \times 100 \quad (2)$$

where

B = bias (percent)
 $PM_{2.5\text{Sampler}}$ = $PM_{2.5}$ concentration as measured by sampler ($\mu\text{g}/\text{m}^3$)
 $PM_{2.5\text{Audit}}$ = $PM_{2.5}$ concentration as measured by audit device ($\mu\text{g}/\text{m}^3$).

The quarterly average bias for a single sampler (\bar{B}) may also be calculated as:

$$\bar{B} = \frac{1}{n} \times \sum_{i=1}^n B_i \quad (3)$$

where

\bar{B} = average quarterly bias (percent)
 B_i = bias for the i^{th} measurement (percent)
 n = number of measurements during quarter.

13.4 Precision

13.4.1 Overview

$PM_{2.5}$ precision is assessed by collocating samplers (i.e., locating a second sampler alongside the primary sampler used to report a measurement). Both samplers are run identically and the results from both samplers are reported to EPA. The collocated (duplicate) sampler's inlet must be placed horizontally between 2 and 4 m from the reporting sampler and within 1 m of the same height (elevation) as the reporting sampler (Section 5.1). The collocated (duplicate) sampler must

be operated exactly as the primary reporting sampler (for example, it should be operated on the same schedule with the same operators, balance, etc.). It is important to designate one sampler always as the primary or reporting sampler and the other always as the collocated or duplicate sampler, even where the reporting sampler may be identical in make and model to the collocated sampler. Under no circumstances should data from the collocated sampler be reported as coming from the reporting sampler, even to make up for missing data from the reporting sampler. The precision goal is to have a CV of less than 10 percent.

13.4.2 Number of Collocated Samplers Required

To assess precision of their sampling data, SLAMS reporting organizations must meet the following collocation requirements:

- At least one reporting sampler must have a collocated sampler.
- At least one of the collocated samplers must be an FRM sampler (i.e., not a designated equivalent method sampler).
- At least 25 percent of all reporting samplers must have collocated samplers. If fractional numbers of collocated samplers are required, values of 0.5 or greater must be rounded upward to the nearest sampler. For example, a reporting organization with 10 reporting samplers must provide collocated samplers for at least 3 of those reporting samplers.

In addition to these requirements, additional specifications must also be observed with respect to the different makes and models of samplers used as collocated samplers:

- Collocated samplers for FRM-designated reporting samplers shall always be of the identical FRM designation (i.e., the collocated and reporting samplers are generally of the same make and model).
- Half of the collocated samplers for designated equivalent reporting samplers have the identical equivalency designation (generally the same make and model), while the other half be FRM-designated samplers. In cases where odd numbers of collocated samplers are required, the number of collocated FRM samplers always be rounded upward to the nearest sampler, with the remaining number being samplers of the same equivalent designation (generally samplers of the same make or model). For example, if five collocated samplers are required, then three should be FRM models and the remaining two should be models of the same equivalency designation (generally the same make and model).

As an example, consider the minimum collocation requirements for an organization that has 43 reporting samplers composed of 25 FRM samplers, 10 equivalent designation “A” samplers, 2 equivalent designation “B” samplers, and 6 equivalent designation “C” samplers. Each type of sampler should be considered separately as shown in Table 13-1 for sampler types “A,” “B,” and “C.”

Some reporting organizations may participate in both the SLAMS (particulate) and visibility networks. The reporting organization may also use $PM_{2.5}$ samplers in the visibility network; however, the visibility network samplers are not required to be FRM or equivalent-designated samplers. These overlapping networks frequently share one or more $PM_{2.5}$ samplers. For this

TABLE 13-1. EXAMPLE OF COLLOCATED SAMPLER REQUIREMENTS

Sampler type	Number of samplers	× 25 percent	Rounded to nearest sampler	Required collocated samplers	Required collocated samplers by type	
					FRM	Same equivalent type
FRM samplers	25	6.25	6	6	6	NA
Equivalency-designated samplers						
Type "A"	10	2.5	3	3	2	1
Type "B"	2	0.5	1	1	1	0
Type "C"	6	1.5	2	2	1	1
Total for reporting organization	43			12	10	2

reason, the visibility and SLAMS (particulate) networks should be combined when determining the number of required collocated samplers for the reporting organization. However, those visibility network $PM_{2.5}$ samplers that are neither FRM nor equivalent-designated samplers should not be counted when determining the number of required collocated samplers.

13.4.3 Location of Collocated Samplers

Collocated samplers should be placed at those sites that have the highest $PM_{2.5}$ concentrations. Reporting organizations that are new to the SLAMS $PM_{2.5}$ network should place special emphasis on sites likely to be in violation of the NAAQS. Data from other particulate measurement methods, such as the PM_{10} samplers, can be helpful in selecting sites for collocation.

SLAMS reporting organizations that have areas in violation of the NAAQS should place their collocated samplers as follows:

- Reporting organizations with sites reporting $PM_{2.5}$ concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate):
 - 80 percent of the collocated samplers should be located at those sites that equal or exceed 90 percent of the NAAQS (annual or 24-hour, as appropriate).
 - The remaining 20 percent of collocated samplers should be located at sites that report less than 90 percent of the NAAQS (annual or 24-hour, as appropriate).
- Reporting organizations **without** sites reporting $PM_{2.5}$ concentrations exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate):
 - 60 percent of collocated samplers should be located at sites that rank in the top 25 percent of the highest sites for $PM_{2.5}$ concentrations (annual or 24-hour, as appropriate).

- The remaining 40 percent of collocated samplers should be located in the remaining 75 percent of sites.

13.4.4 Schedule for Operation of Collocated Samplers

Collocated samples should be taken to reflect the normal operation of the reporting sampler. The collocated samples should be evenly distributed across the various seasons and days of the week. A recommended simple system to accomplish this is to run the collocated sampler every sixth day. Thus, if the first week's collocated sample was run on Monday, the second week's collocated sample would be run on Sunday, the third week's on Saturday, etc. Remember to start and stop both the collocated and reporting samplers at exactly the same time for each sample run.

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Appendix A—Measuring Alkalinity of Filters

A.1 History

Alkalinity of hi-vol filters is measured because alkaline sites react with SO₂, NO_x and other acid gases in the air forming sulfates, nitrates, etc. Such compounds' masses are indistinguishable from particulate mass collected by the filter. In turn, the total particulate weight, sulfate, content, nitrate content, etc., would be falsely high in proportion to filter alkalinity.

A.2. Scope and Applicability

This method describes procedures for measuring the alkalinities of glass and quartz fiber filters. New filters are tested for compliance with EPA alkalinity specifications and older filters are tested in order to evaluate appropriate correction factors for previously obtained particulate data. Table A-1 outlines the procedure.

A.3 Equipment and Reagents

Table A-2 lists the equipment and reagents. The supplier is always Fisher Scientific Company, unless otherwise specified.

TABLE A-1. GENERAL DESCRIPTION OF EPA FILTER ALKALINITY PROCEDURES

Parameter	EPA specification
Sample container	250-mL Phillips beaker
H ₂ O extraction volume, mL	100
Heating technique	Hotplate, reflux condenser (sample temperature about 100 °C)
Extraction time, min	20
Mixer	Magnetic stirrer with 2 in. stirring bar
Filtration equipment	7-cm ID unheated Buchner funnel 250-mL filter flask
Filtration procedure	Twice through Whatman 41 or 541
Titration vessel	250-mL filter flask (no transfer needed)
Titration technique	Potentiometric without temperature control or exclusion of atm CO ₂
Endpoint	pH = 7.00 ± 0.01 after 5 min equilibration (electrode stationary)

TABLE A-2. EQUIPMENT AND REAGENTS

Minimum Qty	Description	Supplier, Cat. No.
Equipment		
1 roll	Heavy duty aluminum foil or stainless steel surface	01-213-11
1 pair	Scissors or shears	14-275C
1 pair	Forceps, 300 mm in length	10-316C
1	Ruler, 12 in. (300 mm) hardwood or metal	12-090
2 pairs	Lint-free gloves	11-393-5
1	Analytical balance (accurate to 1 mg)	
100	Aluminum weighing dishes	08-732
1 pair	Heat-resistant gloves	Lab Safety Supply H1955-2
1	pH Meter (accurate to 0.01 pH units)	
2	Combination pH electrode with AgCl reference and ceramic junction	
2	Electrode storage containers	13-640-300
1	Rectangular porcelain stand	14-667
1	Double buret clamp for above	05-779
2	Buret, 10 mL graduated in 0.05 mL, Class A	03-700-22A
1	Buret, 25 mL or 50 mL graduates in 0.1 mL, Class A, 3-way stopcock optional	03-724-10A
3	Beakers, 10 mL or buret covers	02-539C
2 (optional)	Aspirator bottle with tubulation, 1,000 mL (storage reservoir for H ₂ SO ₄ titrant)	02-972D
3	Volumetric flasks, 1,000 mL, Class A	10-210G
1	Volumetric flask, 500 mL Class A	10-210F
1	Pipet, 1.00 mL, Class A	13-650-2B
1	Pipet, 4.00 mL, Class A	13-650-2E
1	Pipet, 10.00 mL, Class A	13-650-2L
1	Pipet, 100.00 mL, Class A	13-650-2U
1	Extension-type ring, 2–3 in. or clamp	14-055B
2	3-prong clamp, 57 mm grip size	05-740
3	3-prong clamp, 89 mm grip size	05-742
6	Clamp holders	05-757 or 05-759
1 or 2	Ring stands	
1 or 2	Lead bricks for above (or similar weighted object)	
1	Hotplate/stirrer, Corning PC-351 or equivalent	11-495-50
2	Hotplates, Corning PC-35 or equivalent	11-495
1	Watch glass, 90-mm diameter	02-612C
1	Erlenmeyer flask, 2,000 mL	10-040M

(continued)

TABLE A-2 (continued)

Minimum Qty	Description	Supplier, Cat. No.
1	Neoprene or rubber stoppers #6 ½, with hole to fit Buchner funnel	14-141H
2 (optional)	#8, for aspirator bottle	14-141K
1	#7, with hole to fit condenser	14-130L
1	#7, with hole to fit tube below	14-130L
1	1/4 in. to 3/8 in. O.D. heavy-walled glass or metal tube approximately 5 to 6 in. long	
As needed	Vacuum tubing	
	Rubber	14-173C
	Tygon	14-169-2B
1 (2nd optional)	Tubing connector	
	Y-type	15-320-10C
	T-type	15-319C
1	Vacuum source (house vacuum, water aspirator, etc.)	
1	Buchner funnel for 7 cm filter paper	10-356C
1	Box of filter paper, Whatman 41 (or 541), 7 cm	09-850A
1	Forceps, 150 mm L	10-275
1	Stopwatch or equivalent timer, accurate to 1 sec. or better	14-648 or 14-649-5
1	Spatula	
2	Wash bottles, polyethylene	03-409-10E or 03-409-22C
2	Tall form beakers, 200 mL or 150 mL beakers	02-545B
1	Graduated cylinder, 100 mL	08-549-5E
9	250 mL Phillips beakers or 250 mL wide mouth flasks	02-566B
10	Filter flasks, 250 mL	10-181D
Various	Assorted stir bars Teflon® coated or glass beads	10-181D
1	Magnetic stir bar 2 in. LONG, Teflon coated	14-511-65
1	Beaker, 400 mL	02-540L
1	Water-cooled condenser, must be fitted to Phillips beakers or wide mouth flasks above	07-723B
1	Filter flask 500 mL	10-180E
As needed	Laboratory tissues and towels, sizes as available	
1	Box glassine envelopes, #7 4 1/8 in. x 6 1/4 in., ungummed flap	Worcester Envelope Co., #3525
1	Face shield	11-409-5
1	Box hose clamps	American Scientific Products, C6096-232

(continued)

Minimum Qty	Description	Supplier, Cat. No.
Reagents		
1	Case pH standard buffer solution, pH 7.00	SB107-500
1	Case pH standard buffer solution, pH 10.00	SB115-500
1	0.0500 N, Sodium Hydroxide NaOH, Fisher CERTIFIED, C.A.S. Reg. 1310-73-2, 1-L bottle	SS278-1
1	1.000 N, Sulfuric Acid, H ₂ SO ₄ , Fisher CERTIFIED, C.A.S. Reg. 7664-93-9, 1-L bottle	SA212-1
1	Potassium Chloride, CKI, Fisher CERTIFIED, Crystals, C.A.S. Reg. No. 7447-40-7, 500-g bottle	P-217-500
1	Hydrochloric Acid, HCl, ACS reagent, C.A.S. Reg. No. 7647-01-0 ½ gal bottle	A-144-212
	Deionized water	Laboratory Deionization System

A.4 Procedure

A.4.1 Sample Preparation

Filters should be handled only with clean lint-free gloves or clean metal utensils such as forceps, tongs, scissors, etc.

- (1) After initial receipt, place the Hi-vol filters in a metal rack with separate slots for each filter. Place this rack in a controlled humidity (< 50% relative humidity), controlled temperature (25 °C), balance room and allow the filters to equilibrate for 24-48 hours. At the end of that period, carefully weigh each filter using a balance adapted for this purpose. Weigh to the nearest milligram or better. Then place the filters in a storage container.
- (2) Remove one filter at a time from the storage container.
- (3) Place the filter on a clean metal surface (aluminum foil spread on a regular lab bench, clean stainless steel surface, etc.). Measure its length and width on all edges. Calculate the area by multiplying the average length by the average width. Cut out the filter number printed in the upper right corner, in a single rectangular piece. Save the number and place it in a glassine envelope.
- (4) Using a ruler, scissors, and lint-free gloves, cut the filter into ½ in. squares. This makes approximately 320 squares.
- (5) Mix the squares and randomly select a sample of 70-80 squares (approximately 1 g). Do not select and analyze the piece of filter containing the filter number. Weigh the filter sample in a tared disposable aluminum weighing dish. The final sample weight must be within one square of 1 g. Record the weight to the nearest milligram.
- (6) Store the remaining squares in a glassine envelope with the filter number showing. NOTE: Filters may be cut a few days before analysis and stored in glassine envelopes.

- (7) Transfer the weighed sample into a 250-mL Phillips beaker or a 250-mL wide-mouth flask. Cover the beaker or flask with the aluminum weighing dish and seal.
- (8) Clean the gloves, utensils and work area of any filter fibers or lint before taking out the next filter.
- (9) Repeat Steps 2-8 until seven Phillips beakers containing filter squares have been prepared. Leave two Phillips beakers empty as blanks. Label the Phillips beakers and 250-mL filter flasks consecutively from 1 to 9 and pair by number for each sample or blank.

A.4.2 Solution Preparation

- (1) Prepare 0.005 N NaOH titrant by diluting a certified 0.0500 N solution 1:10 with degassed deionized water (i.e., pipet 100.0 mL of certified NaOH into a 1,000-mL volumetric flask, dilute to the mark, and mix well). Degas the deionized water by bubbling N₂ through 1 L of water at a rate of approximately 1 L/min for 1 hour. Take care to protect the solution from the air during storage in order to prevent carbonate formation.
- (2) Prepare 0.0100 N H₂SO₄ titrant by diluting certified 1.000 N H₂SO₄ titrant by diluting certified 1.000 N H₂SO₄ solution 1:100 with deionized water (i.e., pipet 10.00 mL into a 1,000-mL volumetric flask, dilute to the mark with deionized water, and mix well).
- (3) Prepare 0.1 M KCl/0.0001 M HCl electrode storage solution by the following process. First, pipet 1 mL of reagent grade concentrated HCl into a 500-mL volumetric flask, dilute to the mark with deionized water, and mix well. Second, place 7.456 g of KCl in a 1,000-mL volumetric flask, add approximately 500 mL deionized water, swirl to dissolve the KCl crystals, pipet 4 mL of the diluted HCl solution into the flask, dilute to the mark with deionized water, and mix well. The solution may be stored in the volumetric flask or similar glass container.

A.4.3 General Notes and Pre-run Setup

- (1) Set up the titration stand and burets well in advance of sample analysis. Place one buret clamp on the titration stand and adjust the clamp height. In the right-hand buret holder, place a 10-mL buret. Check to see that the stopcock is closed. Fill the buret with 0.005 N NaOH solution and place a small beaker or buret cover on top of the buret:

CAUTION

The NaOH solution should not be stored in the buret for any length of time due to its tendency to form carbonates when exposed to the air.

Place the other buret in the other side of the holder, fill the buret with 0.01 N H₂SO₄, and cover the top of the buret with a small beaker or buret cover.

- (2) (Optional: If using buret with 3-way stopcock.) Attach one end of a length of acid-resistant, flexible tubing to the curved tube, A, at the bottom of the buret and connect the

other end to a 3-way tubing connector. Securely fasten both ends using hose clamps. Attach the other two branches of the tubing connector with flexible tubing to the tubes at the base of the aspirator (reservoir) bottles. If a single reservoir bottle is used, A may be attached directly by a single line. Again, secure the connections with hose clamps. Check the buret stopcock and make sure it is closed. Fill the reservoir bottle(s) with 0.0100 N H_2SO_4 and fill the buret by adjustment of the 3-way stopcock to the proper position. During analysis, cap the reservoirs with beakers. For overnight and longer storage, stopper the bottles with the appropriate sized stoppers. A regular 25- or 50-mL buret may also be used with a Nalgene self zero buret kit #364-2550.

- (3) The filtration system should also be set up in advance. Clamp a 500-mL filter flask (serves as a trap) to a ring stand stabilized by a lead brick. Attach a piece of vacuum tubing to the vacuum source and to a 3-way connector. To one branch of the connector attach a short piece of vacuum tubing leaving the end open. Another piece of vacuum tubing must connect the remaining branch of the connector with the side arm of the 500-mL filter flask. Insert a 5-6 in. heavy-walled tube through the one-hole #7 stopper. Seat the stopper with tube in the flask and attach a piece of vacuum tubing to the tube. Connect an extension ring support to the stand (used for holding the Buckner funnel when not in use). Insert the Buchner funnel through a #6.5 neoprene stopper, set it in the ring stand, and cover the funnel with a 90-mm or larger watch glass.
- (4) Insert the lower end of a condenser into a filter adapter or stopper that fits the Phillips beakers snugly. Connect the water supply to the bottom side arm of the condenser using a length of tubing. Also attach a drainage line (which runs to a sink) to the upper side arm of the condenser.

CAUTION

Securely fasten all connections using hose clamps, condenser hose clips or wire, to prevent the hoses from coming loose, causing flooding (especially dangerous around electrical equipment).

Clamp the condenser to a ring stand, turn the water on, and adjust the flow rate.

- (5) At the beginning of each day's analysis, fill the 2,000-mL Erlenmeyer flask with deionized water, add a stir bar, place a watch glass over the mouth of the flask, and set the flask on a hotplate.

WARNING

A stir bar or glass bead must be placed in the flask to prevent violent bumping.

Set the heat on high until the water begins to boil. After boiling starts, turn the heat down sufficiently to just maintain boiling. Also, at this time set a hot plate/stirrer by the ring stand

with the condenser and adjust the heat setting to high. Set the third hotplate (to be used during the cleanup step) in a convenient place and set on high heat. Generally, a 45- to 60-min warmup period is required for the water and hotplates.

A.4.4 pH Meter Calibration and Electrode Storage

- (1) Calibrate the pH meter at the beginning and end of a run as described in the instrument manual and record the calibration data.
- (2) Store electrodes in the 0.1 M KCl/0.0001 M HCl solution described in Section 4.2, Step 3. For short-term storage and between samples the electrode may be placed in a beaker containing approximately 100 mL of storage solution. For long-term storage (weekend or longer) slip the electrode into an O-ring sealed electrode storage bottle approximately three-fourths full of storage solution and tighten the cap to seal the electrode body to the bottle.

A.4.5 Analysis of Glass Fiber Filters

WARNING

Protective equipment must be worn to prevent serious burns.
The sample is hot and caustic and will adhere to the skin.

- (1) Clamp a clean, 250-mL filter flask to the filtration stand and connect the tubing from the vacuum trap to the flask side arm. Set the Buchner funnel on top of the filter flask, place a single 7-cm Whatman 41 or 541 filter circle in the funnel using tweezers, and cover the funnel.
- (2) Set the first Phillips beaker, containing filter squares, near the hotplate/stirrer and add a 2-in. magnetic stir bar.
- (3) Pour 100 mL of boiling water from the 2-L flask into a graduated cylinder. (Use heat-resistant gloves to handle the hot glassware.)
- (4) Set aside the aluminum weighing dish cover, place the Phillips beaker on the hotplate, and turn on the stirrer.
- (5) Add the 100 mL of water to the Phillips beaker, begin timing the extraction, quickly lower the condenser into the beaker and clamp loosely.
- (6) At approximately 19 minutes and 30 seconds after the start of the extraction, don the face mask, turn on the vacuum, and put on heat-resistant gloves.
- (7) At about 19 minutes and 55 seconds quickly lift the condenser out of the Phillips beaker and clamp in a raised position.

NOTE

The stirrer may be stopped at this time.

- (8) At exactly 20 minutes, using heat-resistant gloves, remove the Phillips beaker from the hotplate, swirl, and pour the contents rapidly, but carefully, through the Buckner funnel. Set the beaker on the bench and apply vacuum until the filter pad is just dry on top. The unconnected length of vacuum tubing may be used to regulate the vacuum strength by placing a finger over the hole in the tubing or by leaving it open.
- (9) Remove the vacuum tubing and the funnel from the filter flask. Pour the hot extract back into the Phillips beaker carefully washing down any residual pulp. Reclamp the filter flask, replace the funnel, and reattach the vacuum tubing. Swirl and pour the extract through the filter. Apply vacuum as needed.

NOTE

Steps 8 and 9 should be completed in 2 minutes or less. Typical times have been 1 minute and 30 seconds to 1 minute and 50 seconds.

- (10) Remove the fact shield, detach the vacuum tubing, set aside the Buckner funnel, place the filter flask on the titration stand, and remove gloves.

NOTE

Before titrating the extract just finished, a more efficient use of time may be made by starting the next sample at this point as follows: Remove the stir bar and wipe it clean of filter material adhering to it. Immediately start another extraction by performing Steps 2 through 5 on the next sample.

- (11) Rinse the pH electrode with deionized water, shake off excess, pat dry with a laboratory tissue, and place in the filter flask. Push pH button (meter in Manual Temperature Compensation mode set to 25°) and take an initial pH reading; this reading should be between pH 9 and 11 for glass filters and between 7 and 9 for quartz. Swirl the flask and begin titration.
- (12) Titrate samples with 0.0100 N H₂SO₄. Add the titrant in increments of several milliliters at a time until the pH drops below 8. At that point slow the addition rate to smaller increments of 0.1–0.5 mL of titrant. Stop the titrant flow when the pH reaches 7.00 and stop swirling; the pH should rise by 0.1–0.2 units. If the final calm pH is greater than 7.00, add enough titrant to compensate. If the titration cannot be stopped at pH 7.00 ±0.02, record the calm pH and the volume of titrant for points around pH 7 and calculate the extract volume of titrant required to reach pH 7 by linear regression or extrapolation methods.

A.4.6 Analysis of Quartz Fiber Filters

In order to reduce background alkalinity retained in the funnel, rinse the Buckner funnel once with boiling water before each analysis of a quartz filter. The alkalinity of quartz filters is more than an order of magnitude lower than that of glass filters; therefore, a lower background is required.

- (1) Clamp in place a clean, dry, 250-mL filter flask and a 250-mL filter flask labeled “rinse.”
- (2) Seat the funnel on the rinse flask.
- (3) Fill a graduated cylinder with 100 mL boiling deionized water, pour the water through the funnel and cover the funnel with a watch glass.
- (4) through (9)
Same as Steps 1-5 for glass fiber filters.
- (10) During the extraction, remove the cover from the funnel, pat the funnel bowl and tip dry with laboratory tissues, move the funnel to the clean dry flask, using forceps place a 7 cm Whatman 41 or 541 filter circle in the funnel and recover. Remove the rinse flask and drain.
- (11) through (17)
Same as Steps 6-12 for glass fiber filters.

A.4.7 Analysis of Blanks

Blank flasks are empty flasks that do not contain any filter squares.

- (1) through (11)
Same as Steps 1-11 for glass fiber filters.
- (12a) FOR BASIC BLANKS: If the initial pH is above 7, titrate slowly with the H_2SO_4 used for samples. See Step 12 for glass fiber filters.
- (12b) FOR ACIDIC BLANKS: If the initial pH is below 7, titrate slowly with the 0.005 N NaOH from a 10-mL buret, instead of H_2SO_4 . Slow the titrant flow when the pH goes above 6 and stop the flow when pH reaches 6.8–6.9; when swirling is ceased the pH should rise 0.1 to 0.2 units. See Step 12 for glass fiber filters.

A.4.8 Cleanup after Analysis

- (1) Wipe and rinse the stir bars, then place in a clean dry place.
- (2a) FOR GLASS FILTERS: Get the next clean filter flask, mount it on the ring stand, and attach the vacuum line. Remove the used filter with forceps, clean the funnel with laboratory tissues, seat it on the filter flask, add a fresh Whatman 41 or 541 filter with forceps, and cover the funnel.
- (2b) FOR QUARTZ FILTERS: Mount the next filter flask and the rinse flask on the ring stand. Attach the vacuum line to the filter flask. Remove the used filter with forceps, clean the funnel with laboratory tissues, seat the funnel on the rinse flask and cover.
- (3) After each titration, rinse the pH electrode into a waste beaker, then with a laboratory tissue wipe off any condensation on the electrode, and place it in the storage beaker.

- (4) As time permits, rinse the Phillips beakers and filter flask thoroughly with a stream of deionized water. Then fill each twice with fresh deionized water discarding the water each time. Fill each a third time with fresh deionized water, add a small Teflon-coated stir bar or clean glass bead and place on a hotplate to boil (as time and space permit).

WARNING

Be sure to use heat-resistant gloves or tongs when handling the hot glassware to prevent burns.

After boiling for at least 5 minutes, discard the water, taking care not to lose the stir bar, drain, and allow to dry thoroughly before reusing. Once per week, or more often as needed, scrub the sides and spout of the Phillips beaker with a clean brush, then rinse thoroughly and boil as above. Store the flask in a clean dry place.

A.5. Calculations

- (1) If the titration was not stopped at exactly pH 7.00, then the titrant volume corresponding to pH 7.00 is calculated by extrapolation or linear regression.
- (2) Calculate the microequivalents for the samples and the blanks using the following equation:

$$\text{microequivalents} = (\text{mL of titrant}) \times (\text{normality of titrant}) \times 1,000.$$

The sign of the blank (or sample) is negative if the titrant was NaOH and positive if the titrant was H₂SO₄.

- (3) Next average the blanks for the day:

$$\text{average blank} = \frac{\text{blank}_1 + \text{blank}_2}{2} \quad (\text{microequivalents})$$

- (4) Then calculate alkalinity in microequivalents per gram of sample using the following equation:

$$\frac{\mu\text{eq}}{\text{g}} = \frac{\mu\text{eq of sample} - \mu\text{eq of average blank}}{\text{gram weight of sample}}$$

- (5) If units of microequivalents per square centimeter are required:

$$\frac{\mu\text{eq}}{\text{cm}^2} = \frac{\mu\text{eq}}{\text{g}} \times \frac{\text{weight of entire filter (g)}}{\text{surface area of entire filter (cm}^2\text{)}}$$

- (6) Sample calculation

A filter weighing 4.0432 g was found to have an area of 517.2 cm². A 1.005-g filter sample was extracted and the extract titrated with 0.0100 N H₂SO₄. The volume 10.99 mL of titrant corresponded to pH 7.08 and 11.12 mL of titrant corresponded to pH 6.92. Both blanks required H₂SO₄ and had values of 1.0 and 0.8 microequivalents, respectively. Alkalinity is calculated as follows:

Step 1 using linear regression:

The estimated volume of titrant at pH 7.00 in this case equals 11.06 mL; or

Step 1 using extrapolation

The estimated volume of titrant also equals 11.06 mL.

Step 2

$$\begin{aligned}\mu\text{eq} &= (11.06 \text{ mL}) \times (0.0100 \text{ N}) \times 1,000 \\ &= 110.6 \text{ microequivalents}\end{aligned}$$

Step 3

$$\text{average blank} = \frac{1.0 + 0.8}{2} = 0.9 \mu\text{eq} .$$

Step 4

$$\text{alkalinity } (\mu\text{eq/g}) = \frac{(110.6 - 0.9) \mu\text{eq}}{1.005 \text{ g}} .$$

Step 5

$$\text{alkalinity } \frac{\mu\text{eq}}{\text{cm}^2} = \frac{4.0432 \text{ g}}{517.2 \text{ cm}^2} \times \frac{109.2 \mu\text{eq}}{\text{g}} = 0.854 \frac{\mu\text{eq}}{\text{cm}^2} .$$

A.6 Quality Control

Prepare a total of nine Phillips beakers for analysis each day; leave two empty as blanks. The sample analysis order is: two samples, one blank, three samples, one blank, two samples. Two samples should be from filters previously analyzed. Number the Phillips beakers and filter flasks from 1 to 9 and rotate the position each day so all glassware pairs eventually are used for a blank analysis. An example of the glassware and sample order for a week is given in Table A-3.

TABLE A-3. GLASSWARE AND SAMPLE ORDER

Day 1		Day 2		Day 3		Day 4		Day 5	
Glass	Sample								
1	S-1	2	S-8	3	S-13	4	S-18	5	S-23
2	S-2	3	S-9	4	S-14	5	S-19	6	S-24
3	B-1	4	B-3	5	B-5	6	B-7	7	B-9
4	S-3	5	S-10	6	S-15	7	S-20	8	S-25
5	S-4	6	S-1	7	S-9	8	S-16	9	S-18
6	S-5	7	S-11	8	S-16	9	S-21	1	S-26
7	B-2	8	B-4	9	B-6	1	B-8	2	B-10
8	S-6	9	S-12	1	S-17	2	S-22	3	S-27
9	S-7	1	S-5	2	S-1	3	S-13	4	S-4

TABLE 2-1. DEFINITIONS OF KEY TERMS

Term	Definition
Accuracy	The degree of agreement between an observed value and an accepted reference value; includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations estimated by collocated sampling.
Actual conditions	The actual ambient temperature and pressure of the atmosphere over the time of sample collection.
Analyst	A staff member who weighs the new and used filters and computes the concentration of PM _{2.5} in µg/m ³ .
ANSI	American National Standards Institute, administrator and coordinator of the U.S. private sector voluntary standardization system.
ASTM	American Society for Testing and Materials, a professional organization that develops and distributes protocols for testing and provides reference standards.
ASTM Class 1 standards	The standards for weighing operations with a microbalance. Certified by ASTM.
Bias	The systematic or persistent distortion of a measurement process that causes errors in one direction.
CAA	Clean Air Act.
Cassette	A device supplied with PM _{2.5} samplers to allow a weighed Teflon [®] filter to be held in place in the sampler and manipulated before and after sampling without touching the filter and to minimize damage to the filter and/or sample, during such activities.
Class I equivalent method	An equivalent method for PM _{2.5} based on a sampler that is very similar to the one specified for the reference method (40 CFR Part 58, Appendix L), with only minor deviations, as defined by EPA.
Collocation	The placement of a second PM _{2.5} sampler (known as the duplicate sampler) near the first sampler (known as the primary sampler) used to report air quality at the site to estimate precision and bias by comparing the results of the two samplers. The sampler inlets should be no closer than 2 m and no further than apart 4 m.
Control chart	A graphical presentation of quality control (QC) information over time. If a procedure is "in control," the results usually fall within established control limits. The chart is useful in detecting defective performance and abnormal trends or cycles, which can be corrected promptly.
DAS	Data Acquisition System. Often is a computerized system.
Downtube	The perpendicularly oriented tube that connects the PM _{2.5} 's sampler inlet to sampler components inside the sampler case. To check the flow rate, the sampler inlet is removed from the downtube and a flow rate standard is connected in its place.
Dummy filter	A filter placed in the sampler but not used for sampling, but used for leak checks, etc.

(continued)

TABLE 2-1 (continued)

Term	Definition
Electrostatic charge buildup	A buildup of static electricity on an item, such as the PM _{2.5} filter, which makes it difficult to handle and can influence its proper weighing.
Equilibration chamber	A clean chamber usually constructed of plastic or glass, held at near constant temperature and humidity, used to store PM _{2.5} filters until they and their particulate catch have reached a steady state in weight.
FEM	Federal equivalent method for PM _{2.5} sample collection.
Field blank filter	New filters that are weighed at the same time that presampling weights are determined for a set of PM _{2.5} filters. These field blank filters are transported to the site in the same manner as a filter intended for sampling, installed in the sampler, removed from the sampler without sampling, and stored in their protective metal containers inside the sampler's case at the sampling site until the exposed filter(s) is retrieved for postsampling weighing in the laboratory, where it is handled in the same way as an actual sample filter and reweighed as a QC check.
Flow adapter device	A tight-fitting connecting device that is inserted in place of the PM _{2.5} sampler inlet on the sampler's downtube and used to connect flow calibration or audit devices to check the flow rate.
Flow calibration device	A traceable flow determining apparatus (also called a flow rate standard) that is attached to the flow adapter device and used to assist in setting and measuring the volumetric flow of air into the sampler. Bubble flow meters, piston flow meters, and orifice devices are the most common.
Flow check filter	A standard PM _{2.5} filter that is placed in the sampler during the flow check procedure; it is later discarded and not used for further sampling.
FRM	Federal reference method for PM _{2.5} sample collection.
Impactor	An inertial particle size separator. Reference or Class I equivalent PM _{2.5} samplers use specially shaped inlets followed by impactors that separate particulate matter into well-defined size ranges.
Impactor well	That portion of the sampler inlet where particles smaller than 10 µm but larger than 2.5 µm impact and are held by a filter soaked with oil (tetramethyl tetraphenyltrisiloxane).
Laboratory blank filter	New filters that are weighed at the time of determination of the pre-sampling (tare) weight of each set of PM _{2.5} filters intended for field use. These laboratory blank filters remain in the laboratory in protective containers during the field sampling and should be reweighed in each weighing session as a QC check.
Leak check	Checks made to ensure there are no leaks into or within the sampler. The external leak check can be carried out automatically by the sampler. The internal bypass leak check determines if any portion of the flow bypasses the sampler's filter.
Mass reference standard	ASTM- or NIST-traceable weighing standards, generally in the range of weights expected for the filters.

(continued)

TABLE 2-1 (continued)

Term	Definition
Microbalance	A type of analytical balance that can be read to the nearest 0.001 mg (that is, one microgram or one-millionth of a gram).
NAAQS	National Ambient Air Quality Standard.
NAMS	National Air Monitoring Stations.
NIST	National Institute of Standards and Technology. Supplier of the ultimate reference standards and materials in the United States.
NPAP	National Performance Audit Program.
Operator	A staff member who services the PM _{2.5} sampler at the field site and who transports the filters to and from the laboratory.
Orifice flow rate check device	One type of flow calibration or check device based on orifice plate flowmeter principles and often used in the field. The flow rate is determined by relating the pressure drop across the orifice to the flow rate of air established through calibration with a standard such as a soap film flowmeter.
P _a	Ambient pressure.
P _{std}	EPA designated standard barometric pressure, 760 mm Hg.
PAMS	Photochemical assessment monitoring station.
PM _{2.5}	Particulate matter (suspended in the atmosphere) having an aerodynamic diameter less than or equal to a nominal 2.5 µm as measured by a reference method based on 40 CFR Part 50, Appendix L, and designated in accordance with 40 CFR Part 53.
PM _{2.5} sampler	A sampler built to EPA specifications that collects a sample of particulate matter from the air based on principles of impaction and filtration. The sampler also maintains a constant flow and records the total actual flow so that PM concentration can be calculated from the weight of the filter catch. A sampler is not automated to give concentrations directly.
Polonium-210 (²¹⁰ Po) antistatic strip	A device containing a small amount of ²¹⁰ Po that emits α particles (He ²⁺) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.
Precision	A measure of random variation among individual measurements of the same property, usually under prescribed identical conditions. For ambient particulate concentration measurements, precision is always expressed in terms of the standard deviation estimated by collocated sampling when data are reported to the AIRS database.
Primary standard for PM _{2.5}	(a) 15 µg/m ³ , annual mean, based on the 3-year average of the annual arithmetic mean PM _{2.5} concentrations, spatially averaged across an area; (b) 65 µg/m ³ , 24-hour average, based on the 3-year average at the 98th percentile of 24-hour PM _{2.5} concentrations at each population-oriented monitor within an area.
PTFE	Polytetrafluoroethylene, the polymer that makes up the 47-mm diameter filter.

TABLE 2-1 (continued)

Term	Definition
Q_a	The sampler flow rate at ambient (actual) conditions of temperature and pressure.
Q_{std}	The sampler flow rate at EPA standard conditions (1 atm pressure and 25 °C). Not used in calculations to express $PM_{2.5}$ concentrations.
QA supervisor or coordinator	A staff member who assists in preparation of the reporting organization's quality plan, makes recommendations to management on quality issues (including training), oversees the quality system's control and audit components, and reports the results.
Readability	The smallest difference between two measured values that can be read on the microbalance display.
Repeatability	A measure of the ability of a microbalance to display the same result in repetitive weighings of the same mass under the same measurement conditions.
SLAMS	State and local air monitoring stations.
Standard conditions	EPA-designated atmospheric conditions of 1 atm pressure (760 mm Hg) and 25 °C (298 K).
T_a	The ambient or actual temperature.
T_{std}	EPA's standard temperature (25 °C).
Traceable	A term signifying that a local standard has been compared and certified, either directly or by not more than one intermediate standard, to a NIST-certified primary standard, such as a thermometer or standard reference material (SRM).
Transfer standard	A reference standard device (for temperature, flow rate, etc.) that has been compared to a NIST reference standard and is subsequently used to make measurements and/or transfer traceability of other devices.
TSP	Total suspended particulate (matter). Collected by a high-volume sampler.
V_a	The air volume at ambient (actual) conditions of temperature and pressure.
V_{std}	The air volume expressed at EPA standard conditions (pressure = 1 atm; temperature = 25 °C).
WINS	The nickname given to the design of a particle impactor; an acronym for "Well Impactor Ninety Six."